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DEVELOPMENT OF FIRESAFE ELECTRICAL CABLE FOR SHIPBOARD USE: PHASE VIII

M.K.Murthy – S.Das Gupta – S.Johar

The Electrofuel Manufacturing Company Limited

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Abstract

A cable design has been developed which meets not only the electrical performance requirements, but also is compatible with physical property specifications, e.g., flexibility, mechanical strength, and moisture resistance.

A material composition comprising 80wt% KAOWOOL-20 wt% BN fibers has been developed to meet the property requirements of the electrical insulation of the cable. A unique semi-automatic "slurry-tape-casting" process has been developed for the fabrication of the ceramic insulation in the form of a continuous tape. Prototype cable segments, with lengths up to 12 feet were assembled from the insulation tape using Ni and Ni-coated copper electrical conducting wires. Although manual techniques were used for prototype cable fabrication, engineering concepts were developed for continuous assembly.

Prototype cables have been evaluated for their electrical performance, stability, mechanical properties, and moisture resistance. They have satisfactorily met all their target specifications. They have operated under electrical load at 1000°C for more than one hour. Also, they have withstood open-flame burning tests for more than three hours.

Résumé

On a mis au point un modèle de câble qui, en plus de satisfaire aux exigences relatives à la performance électrique, répond aussi aux spécifications physiques telles que la flexibilité, la résistance mécanique et la résistance à l'humidité.

Une composition comprenant 80 % de KAOWOOL[MD] et 20 % de fibres de BN (pourcentages massiques) a été mise au point pour obtenir les propriétés d'isolation électrique requises. On a développé un procédé semi-automatique unique de « coulage en pâte de rubans » pour produire un isolant céramique sous forme de ruban continu. Des segments de câble prototypes, pouvant atteindre jusqu'à 12 pieds, ont été assemblés à partir du ruban d'isolation et de fils électro-conducteurs en nickel ou en cuivre revêtu de nickel. Bien que la fabrication du câble prototype utilise des techniques manuelles, on a mis au point des concepts techniques d'assemblage en continu.

On a évalué la performance électrique, la stabilité, les propriétés mécaniques et la résistance à l'humidité des câbles prototypes. Ils ont atteint toutes les spécifications recherchées de manière satisfaisante. Ils ont été utilisés pendant plus d'une heure sous charge électrique, à 1000 °C. Ils ont aussi résisté aux tests de combustion à la flamme nue pendant plus de trois heures.

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Development of Firesafe Electrical Cable for Shipboard Use: Phase VIII

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EXECUTIVE SUMMARY

Introduction

Conventional electric cables represent a significant fire load onboard ship owing to the inherent combustibility of the polymeric materials used to make the cable sheathing. Moreover, burning cable sheathing materials produce dense toxic smoke and fumes and fire can travel from one compartment to another throughout the ship along burning cable runs. The goal of this work was to develop inorganic materials and processes that could be used to make cable sheathing material that would be less vulnerable to fire involvement and fire damage.

Principle Results

This report describes the design and the materials and process development and testing of a firesafe high temperature electrical cable that was done under a DREA research contract with the Electrofuel Manufacturing Company, Limited. A fireproof inorganic cable sheathing was developed and used to make prototype electrical cables which not only met electrical performance requirements but also had good flexibility, mechanical strength and moisture resistance.

Significance of Results

The work done here proved that inorganic (ceramic) materials could be used to produce fireproof electrical cables that met all the normal electrical requirements and were at the same time flexible, strong, lightweight and moisture resistant. It is suggested that the unique semi-automated slurry-tape-casting process that was developed to make the ceramic felt-like cable sheathing material which was used to produce prototype cable segments up to 4 meters in length offered an engineering concept that could be further developed to produce cables of any desired length.

Future Plans

Due to changing priorities and client support at the present time no work is planned in the immediate future in this important area of research. However, in light of some recent accidents in which the fire performance properties of electrical cables may have been a

contributor it is suggested that further research is required to advance the state of the art in production of light weight, flexible and low cost fireproof electrical cables.

DEVELOPMENT OF FIRESAFE ELECTRICAL CABLE FOR SHIPBOARD USE: PHASE VIII

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1.0 INTRODUCTION

The Electrofuel Manufacturing Company has developed a fire-safe high temperature electrical cable based on boron nitride(BN) fibers which are stable at high temperatures, chemically inert and possess high electrical resistivity properties combined with a novel slurry-tape-casting process technology. This development was financially supported by a research program sponsored by The Defence Research Establishment Atlantic, Halifax N.S. to meet naval shipboard requirements for improving fire safety. The cable also meets technical requirements for a number of industrial and commercial applications.

The electrical cable has been designed to withstand temperatures of up to 1000^{o}C for time periods of at least one hour thus making it useful for most applications. The design and development of this high temperature electrical cable was made possible by the technological and engineering capabilities available at Electrofuel, particularly the availability of boron nitride fibers made by in-house facilities, and expertise and capabilities in the development and manufacture of advanced ceramic materials and products. The scientific advice and support of DREA staff contributed immeasurably to this development.

This report describes progress of research carried out between September 1, 1995 to March 31, 1997 whose objective was to study the improvements to the ceramic felt fabrication process, specifically, development and optimization of slurries containing up to 5 wt.% solids as it relates to homogeneity, flow characteristics, quality, and texture of the insulation felt; identification, selection and evaluation of suitable "moisture-barrier(s)" to enhance stability characteristics of the cable; and to evaluate prototype cables to establish their over all performance. In as much as the work carried out in this final phase of the research program was designed to address solutions to a number of scientific, technological and engineering issues related to the conversion of laboratory-scale process technology to an economically viable manufacturing process, and since this report forms the final "*documentation*" on the over all achievements of the program, it has been formatted as a summary report integrating the results of the present study with experimental data and findings of earlier investigations. As before, the reported activities have been grouped into the following five sections.

- Design and specifications of the Cable
- Materials Development
- Cable Process Development
- Cable Testing and Evaluation
- Summary and Conclusions

2.0 DESIGN SPECIFICATIONS FOR THE CABLE

Successful development of design and specifications of fire-safe cable is ultimately governed by its performance requirements, listed in ***Table 1***. Basic considerations in electrical cable design require the separation of electrical conductors by insulating dielectric materials. Four important criteria have to be addressed: Physical Design Parameters (e.g., shape, size, dimensions); Electrical Parameters (such as voltage, current and insulation requirements); Material Parameters (chemical composition, electrical resistance properties, physical state and availability); and, Process Parameters (i.e., fabrication and assembly of cable). Other things being equal, material and process considerations dominate the decision making process in the selection of the design because of their influence on technical feasibility, manufacturing cost and economic viability.

In early approaches to basic design considerations for high temperature cable a number of alternative concepts were developed and analyzed in terms of material property requirements and process technology. This resulted in ***Design No.1*** shown in ***Figure 1***. This design was selected because of its suitability for the extrusion process, a standard ceramic process technology most cost-effective for commercial production. Detailed experimental studies were carried out on material compositions based on boron nitride, alumina, silica, low temperature inorganic binders and organic polymers to enhance plasticity during the extrusion process as well as to impart flexibility to the cable. The extrusion process was successful in terms of the economics of fabrication of the cable and its electrical properties. However, a detailed analysis of the high temperature performance of prototype cables indicated serious limitations with this approach; specifically, the deleterious contribution of organic polymers towards burning and production of toxic gases. Further analysis and critical assessment indicated the desirability and necessity of developing a new design /materials/fabrication process combination which eliminated the use of all organic polymers without loosing the physical flexibility properties of the cable. ***Design No.2***, shown in ***Figure 2***, meets this criteria.

The development of ***Design No.2*** was made possible by two considerations. First, the possibility of developing ceramic electric insulation medium as a flexible felt, flexibility to be achieved by the use of fibrous materials instead of organic polymers and plasticizing agents. Second, the possibility of fabricating the flexible felt by slurry-tape-casting technique. In order to arrive at a workable design, the following parameters had to be addressed: shape and dimensions of the felt; physical and electrical properties; fire resistant properties; and external shell design. The shapes of the different components were determined by the process requirement i.e., the use of ceramic felts made by the slip casting technique. The two important physical property requirements are strength and flexibility. The cable system does not usually come

under a great deal of mechanical loading. Hence, tensile strengths of the order of 100 MPa and compressive strengths of a similar magnitude were considered adequate to ensure good performance during its installation and operating life. Usually, in use, the cable has to meet the requirements imposed by corners and, in some instances, angles more than 90 degrees may be encountered. This required that the cable be sufficiently flexible i.e., it can be bent to an angle of up to 120 degrees for cable up to 2.2 cm in diameter.

The basic ceramic insulation system comprising boron nitride fibers, alumina powders and silica powders used in ***Design No.1*** was found to be fundamentally satisfactory as an electrical insulation medium even in the absence of organic polymers. However, alumina and silica powders were not suitable in their powder form because they did not impart flexibility properties to the cable. This limitation could be overcome by the selection and use of alumino-silicate fibers in the form of commercially available KAOWOOL (47% alumina and 53% silica) manufactured by Thermal Ceramics Co. Also, the possibility of developing "slurry-tape-casting" process for the fabrication of flexible ceramic felt from the foregoing materials looked technically feasible and promising. The thickness of the insulation (i.e., the ceramic felt) had to be designed on the basis of its electrical properties i.e., its electrical resistance and dielectric strength properties. The electrical resistivity properties of the three ceramic components comprising the insulation felt is greater than 10^{13} ohm-cm and is sufficient for the intended applications. The dielectric strength of the materials are in the range 40-160 volts/mil. Thus, for room temperature applications for various voltages, the thickness requirements were calculated as follows:

120 volt operation.....120/40 = 0.003 inches - 75 microns
600 volt operation ..... 600/40 = 0.015 inches - 375 microns

Using a safety factor of 10, the respective thickness requirements are:

120 volt operation ..... 750 microns or 0.75 mm
600 volt operation ..... 3750 microns or 3.75 mm

Another factor which had to be taken into consideration was the nature and thickness of the metal conductors. Copper electrical conducting wires were selected. The diameter of the wire is governed by the current carrying capacity required. For ratings up to 20 amps, 12-14 AWG wires are to be used. Higher current ratings require thicker wires. A detailed discussion of materials and process development follows in subsequent sections.

Design No.2 formed the basis for most of the research in earlier phases of the program and looked very promising. Prototype electrical cable segments 2 to 6 feet in length were fabricated and were evaluated for performance.

The mechanical properties, particularly flexibility, were found to be satisfactory. The cables maintained their electrical resistivity integrity up to 1000^{o}C in air, however, they deteriorated when the ambient humidity increased to 85%. Since the cables have to perform under severe environmental conditions on board naval ships, including exposure to extreme humidity/moisture conditions, it once again became necessary to identify the problems associated with this design and to address improvements to overcome the limitations.

An analysis of possible mechanisms traced the moisture adsorption/ absorption problems to the high porosity (83 %) of the felt and the very high surface area of the boron nitride and alumino-silcate fibers comprising the felt. As described in a subsequent section, several techniques of coating water repelling silicone materials on the fibers were evaluated without much success, the reason being the important contribution of porosity of the felt. Even though the coated fibers were hydrophobic, the porosity of the felt permitted moisture/water absorption by capillary action. After considering a number of alternative preventive techniques, it was concluded that the best way of overcoming the problem was to introduce a moisture/water repelling polymer (polypropylene) membrane between the fibrous insulation and the high temperature alumina sleeve covering of the cable. This change is shown in ***Design No.3*** shown in ***Figure 3***. In the period of this report, cable segments (2 to 6 feet) have been re-evaluated for their electrical and burning performance characteristics. The cables maintained their electrical integrity for more than one hour at 1000^{o} C indicating that they meet the original technical specification guidelines.

Two important drawbacks for using polypropylene moisture/water barrier in the cable are its contribution to the fire load of the cable, and the possibility of moisture/water migration from junction/switch boxes where the cables are connected. Since polypropylene film forms only about 0.1-0.2 wt.% of the cable, its contribution to the fire load is considered negligibly small and may not be a serious problem for most applications. In any case, its effective contribution can be further decreased by increasing the thickness of the alumina protection sleeve to increase the heat transfer time. For the most critical applications, alternatives exist where additional monolithic ceramic protection can be provided but, at increased cost. Problems associated with moisture/water migration through junction/switch boxes are not considered very serious because they can be protected by the use of monolithic water-impervious ceramics, although at additional cost.

In conclusion, it is felt that ***Design No.3*** offers the best compromise, given the present status of technology development and the efforts expended to date in developing it.

3.0 MATERIALS DEVELOPMENT

3.1 Status

Four different types of materials are required for the fabrication of electrical cables. They are:

- Ceramic electrical insulation
- Electrically conducting wire for electrodes
- moisture barrier films/coatings
- protective ceramic sleeving.

The bulk of the research efforts have been devoted to the development of electrically insulating ceramic felt comprising BN fibers and KAOWOOL fibers because such a product is not available commercially.

Electrically conducting wires were identified for use as cable conductors. In the early part of the program copper wires were used because of their high conductivity and commercial availability in the required gauge sizes. The research activities comprised establishing compatibility with the new insulation felt (e.g.., chemical stability) as well as meeting the technical requirements for high temperature cables in terms of current carrying capacity, flexibility etc. However, serious problems were encountered during the testing and evaluation of cables because the melting point of copper is only 1083^{o}C and is too close to the maximum operating temperature of 1000^{o}C of the cable; furthermore, serious compatibility problems between the ceramic insulation and copper wires were encountered, particularly corrosion and its possible effect on the life of the cable. Alternative conductor materials were evaluated and, based on commercial availability, Ni-clad copper and pure nickel wires were selected. Both the wires survive 1000^{o}C for one hour, the specification for the cable under development, however, at this temperature the life of a pure nickel conductor is much higher. Because it is cheaper, nickel coated copper is preferable unless application demands justify the higher cost of pure nickel wires.In this program 16 gauge nickel wire supplied by the A.D.Mackay Company has been used.

Moisture barrier materials are required to protect the electrical cable from the deleterious influence of moisture/water, a common environment encountered in naval applications. Research on moisture barrier materials has comprised studies on two types of materials, their application, and evaluation: first, liquid material compositions that can be used either to impregnate and/or coat the insulation felt; secondly, on polymer films/sheets which can be used to wrap around the cable. The second approach appears to be more promising. Research on moisture barrier materials was extensive and essentially consisted of the identification of suitable products and their performance evaluation as an

integral part of the cable. A detailed discussion of the research activities is described in a subsequent section.

Protective ceramic sleeve material was identified from commercial sources and consisted of a woven product made from high purity alumina fibers.

The impetus for undertaking the cable development program by Electrofuel was the availability of in-house manufacturing capabilities for boron nitride (BN) fibers and their unique properties as an electrically insulating material. The properties are listed in ***Table 2***. Boron nitride has very high electrical resistivity, very high chemical inertness and stability at high temperatures. Although BN fibers could have been used alone as a complete insulating material, in practice it was necessary to incorporate other suitable materials not only to reduce cost but also to promote sinterability of the material system to achieve the required densification and mechanical strength. Alumina (Al_2O_3) and silica (SiO_2) were identified as suitable ceramic materials because of their high electrical resistivity properties, as well as their ability to withstand high temperatures and corrosive environments. Early in the program, the sintering and electrical properties of the binary system BN-Al_2O_3, and ternary system BN-Al_2O_3-SiO_2 were studied to identify and establish the most suitable compositions for cable applications. Material compositions comprising 20-22 wt.% of BN, 48-52 wt. % of alumina and 12-14 wt. % silica, the latter two in the form of ceramic powders were found to meet the needs in terms of electrical insulation and mechanical properties. In fact, these compositions with the required amounts of polymers to achieve extrudability and flexibility formed the basis for "extrusion process technology" developed to fabricate cables of ***Design No.1***. With the development of ***Design No.2***, it became necessary to replace alumina and silica powders with fibers to achieve flexibility properties of the cable fabricated by the "slurry-tape-casting" process. Although fibers of these material compositions are commercially available, they are prohibitively expensive because they are manufactured using high temperature processes. Hence, it was decided to use commercially available alumino-silicate fibers which are relatively cheap because of their extensive use in the manufacture of refractory insulation products.

Three commercial alumino-silicate fibers were identified as possible candidates: FIBREFAX and FIBREMAX manufactured by Carborundum Company and KAOWOOL manufactured by Thermal Ceramics. Their chemical compositions and costs are listed in ***Table 3***. Although all three are basically alumino-silicates, their chemical compositions are significantly different. KAOWOOL contains the least amount of alumina(47%) and the highest amount of silica(53%) and, therefore, is relatively more reactive; FIBREMAX contains the largest amount of alumna(72%) and the least amount of silica(27%); FIBREFAX falls in between with 51.9% alumina and 47.9% silica.

Since the basis for the "slurry-tape-casting" process is a slurry comprising the starting materials, a systematic study of the rheological properties as well as mixing and blending parameters, particularly viscosity and pH values of water slurries containing different amounts of the above three alumino-silicate fibers were carried out to delineate their behavior and select the most suitable composition for cable use. Because of their fibrous nature, these alumino-silicates have very high surface areas and react with water. The pH values of the slurries have been used as a measure of this reactivity. Viscosity of the slurry is an important parameter because it governs not only the amount of fibers in the slurry (i.e., solids content) but also controls its flow properties which are critical from a processing point of view. The experimental details are described in some detail because of their importance; similar studies were used extensively in the subsequent technology development program and formed an important source of useful experimental data required for process development.

The pH and viscosities of the slurries were determined using the following procedure. Various weight percentages of the alumino-silicate fibers were mixed in 500 ml. of distilled water in a high speed blender. Slurries were characterized by measuring the pH, settling time and viscosity. Viscosity in centipoise (cps) was determined using an LV series Brookfield viscometer having an LV1 spindle at a shear rate of 30 RPM. Both the viscosity and the pH of the slurries have a bearing on the settling rate of the solids from slurries.The experimental data for pH and viscosity are listed in ***Table 4*** and summarized in ***Figures 4*** and ***Figure 5***, respectively. The pH values for KAOWOOL are in the range 7.7-8.4 compared to FIBREFAX and FIBREMAX, which are in the range 5.4-6.4, indicating that it is more reactive. A comparison of the viscosity values of the three materials indicated that both FIBREMAX and KAOWOOL have low values in the range 65-140 cps. After an evaluation of the results, KAOWOOL was selected as the best material for the cable development program because of its higher reactivity and low cost ($3.70/lb) compared to FIBREFAX ($13.33/lb) and FIBREMAX ($34.25/lb).

3.2 Current Work

Much of the research work on materials in the current phase of the program was devoted to a better understanding of: the rheological properties of slurries containing solids of composition 80 wt.% KAOWOOL-20 wt.% BN fibers; the properties of ceramic insulation felt, particularly physical, mechanical and electrical performance; and the identification and evaluation of moisture barrier films. Also, commercial information on these materials has been acquired.

4.0 CABLE PROCESS DEVELOPMENT

Cable development comprises three independent process steps:

- Development and Evaluation of Slurries
- Process Development for the Ceramic Insulation Felt by the Slurry-Tape-Casting Technique
- Process Development for Cable Assembly

4.1 Development and Evaluation of Slurries

Slip casting technology i.e., casting of slurries comprising clays into gypsum molds is routinely used by the ceramic industry in the manufacture of pottery and sanitaryware products. This technology, after suitable modifications, formed the basis for the development of the "slurry-tape-casting" process, the fundamental modification comprising the use of an innovative vacuum-casting technique for the removal of water (the suspension medium in this case) from the slurry instead of gypsum molds.

From a process technology point of view, the most important property criteria that a slurry has to meet are homogeneity of composition, stability, and flowability: Homogeneity of composition means that the solid particles must be uniformly distributed throughout the slurry so as to maintain overall chemical composition: Stability means that the particles in the slurry should stay in suspension with the aid of a suitable mixer and not settle down with time producing a two phase system: Flowability means that the slurry must be able to flow easily as required by the process parameters and conditions. Viscosity and pH parameters can be used as a means of evaluating the foregoing properties. These properties, in turn, are controlled by the nature of the materials used, slurry media, and solids content. Since water is to be used as the slurry media, changes in pH values of slurries can be used to indicate reactivity of the materials. Viscosity is a function of solids content and, to some extent, controls the amount of solids that can exist in homogeneous suspension in a slurry. Particle sizes of the solid materials and their distribution also have an effect on the stability of the slurry; the smaller the particle size the more stable the slurry.

During earlier evaluation studies on electrical properties of materials for cable applications it had been established that a composition comprising 20 wt.% BN fibers and 80 wt.% of a mixture of alumina and silica powders will adequately meet the insulation requirements of the cable under development. Hence, material composition for slurry development was arrived at as 20 wt.% BN fibers and 80 wt.% KAOWOOL fibers, the latter replacing alumina and silica

powders. As a first step, the pH and viscosity properties of slurries containing 5-10 wt.% solids of this composition were studied to understand the influence of BN fibers, if any, on pure KAOWOOL slurries. The experimental results are shown in ***Figure 6***. The results, when compared to earlier results shown in ***Figures 4*** and ***5***, indicate that BN fibers do not influence the viscosity properties significantly; however, the pH values are higher probably because of the presence of residual boric oxide in the BN fibers.

Earlier work in the program on compositions containing 1-10 wt.% solids lead to the development of an optimum slurry with 5% solid materials of a composition comprising 80 wt.% KAOWOOL and 20 wt.% BN fibers. Establishing the optimum solids content of the slurry from a processing point of view turned out to be more complicated than expected. In conventional ceramic practice the solids content of the slurry is kept at the maximum possible concentration level consistent with casting requirements i.e., homogeneity, stability, flowability and minimum casting time: Normally the solids content is in the 25-35 wt.% range. However, since the "slurry-tape-casting" process is new, the specific requirements are not known. Nevertheless, an analysis indicated that two opposing forces are at play; and, that it is necessary to achieve an optimum balance between these two opposing requirements. Because the slurry has to pass through comparatively narrow transfer tubes and a feeder nozzle, it is desirable to have as low a solids concentration as possible to achieve the required low viscosity and high flowability properties. However, the solids content should be high enough to achieve settling in the casting mold in the minimum time required to obtain the required thickness. Satisfying these needs required a systematic and detailed evaluation of the rheological properties of slurries containing KAOWOOL and BN fibers. A brief description of the work follows not only because of its fundamental importance but also because these experimental procedures have been subsequently used throughout the program, either as a control tool to evaluate the starting materials (from batch to batch) and/or for the verification and confirmation of results.

As pointed out in Section 3.1, BN fibers and KAOWOOL fibers were chosen as the staring materials for the fabrication of electrical insulation by the "slurry-tape-casting" process. As a first step, the viscosity and pH properties of slurries containing 5 to 20 wt.% KAOWOOL were studied as a function of solids content. The data are listed in ***Table 4*** and plotted in ***Figures 4 and 5***. The results showed that the maximum solids content for a workable slurry is 10 wt.% (viscosity 140 cps); and that at 20 wt.% solids homogeneity cannot be achieved. The pH values increase slowly as a function of solids content. Viscosity remains almost constant up to 10 wt.% solids, and increases steeply thereafter to reach a value of 1320 cps at 20 wt.% solids. Based on these results additional investigations were carried out to establish more precisely the maximum solid content acceptable from a processing point of view. Detailed experiments were

carried out on slurries containing 2, 4, 6, 8, and 10 wt.% solids. Viscosity and settling properties were measured. The experimental data are presented in ***Table 5*** and ***Figure 7***. As is to be expected, the viscosity of the slurries increased rapidly with increase in the solids concentration; the exponential growth rate implies that for concentrations above 5-6 wt.% the slurries will be too viscous from a processing point of view. Results on the settling properties indicated a 5-2 minute range, decreasing with increasing solids concentration. Based on these results it was concluded that the best solids content for the slurry is 5 wt.%.

From a processing perspective, the settling properties of slurries as well as fiber length distribution are very important and critical. The insulation felt /tape is formed by the removal of water from the slurry in the moving casting mold using a screen mesh. The removal of water has to be accomplished in a very short period of time, usually in seconds. Hence, considerable effort was spent in the early part of the program to establish the settling properties of slurries. Slurries with solids content in the range 0.5 to 3.0 wt.% were studied. The experiments were performed on 1 litre of slurries contained in a 1 litre glass measuring jar. The solids were allowed to settle from a well stirred slurry and the "meniscus" separating the clear supernatant liquid from the solids was used as a measure of settling. The results are plotted in ***Figure 8***. Projection of data for 5 wt.% solids concentration indicates that complete settling takes place in less than 60 seconds ***(Figure 9)***. It was also found that the settling characteristics were sensitive to pH. Fiber length distribution in the slurry is a function of mixing time. ***Figure 10*** shows that BN fiber length decreases rapidly as a function of mixing time. The average length of BN fibers was determined using the optical microscope technique. For mixing times over 15 seconds the average length falls below 6 mm. To maintain longer fiber lengths it is necessary to keep mixing times to a minimum consistent with other requirements. These considerations were immensely useful in establishing the design and speed of the moving carriage of the casting mold, optimum slurry mixing times and other operating parameters.

In order to improve the handling capabilities of the insulation felt/tape, efforts were made to increase their strengths by the incorporation of binders. Attempts to increase felt strength by incorporating binders such as ABS and polycarbonate by impregnation techniques were not completely successful; although strength increased, the felts became brittle. Success, however, was achieved by the use of a 3M Company product (#30 binder). Due to its water solubility it was added to the slurry, resulting in its uniform distribution in the felt. The strength of the felt increases with increase in binder content. However, to limit the degrading properties contributed by the binder to the high temperature properties of the felt, and its contribution to fire load, a compromise concentration of 3% was established. The felt produced in this way was found to

be flexible and water repellent. It did not rupture when folded and could be bent and rolled easily.

In the current phase of the program, all the research efforts have been devoted to identify, establish, and optimize parameters that govern large scale preparation of homogeneous, stable, and flowable slurries containing 5 wt.% solids compatible with the requirements of the new automated tape casting system described in the next section. Since the new slurry holding tank associated with the new facility has a capacity of 100 liters, considerable effort was invested to develop this capability. As appropriate, earlier experiments carried out with smaller volumes of slurry (5-10 liters) were repeated and formed the backbone for the new investigations. A series of experiments were carried out increasing the slurry volumes gradually to 20 liters, 75 liters and 90 liters and the requirements were successfully established.

All experiments were carried out using a 100-liter plastic holding tank. The mixer used in earlier small scale experiments was found to be inadequate and unsuitable for mixing and homogenizing the larger volumes. A special mixer (described in Section 4.2.3) was used. With the new set up, the 20 liter volume needed only 10 minutes to achieve homogeneity. However, for 90 liter volume, 20-25 minutes were needed to achieve the desired homogeneity. Homogeneity was determined by removing small samples at regular intervals and testing them under a light source; inhomogeneities appeared as clumps. Settling time studies showed that the rate of settling did not depend on the volume of the slurry, but it was found to be related to the surface area of the container rather than the volume. Viscosity was measured using a Bohlin Visco 88 Viscometer. Several experiments were performed using optimum conditions, and the parameters measured and typical results are listed in ***Table 6***.

Experiments were also performed to study the variation of slurry properties as a function of time. Two different aspects were studied: the slurry was left without stirring; and, the slurry was continuously stirred. The purpose of these studies was to determine, the influence of chemical reactions, if any, between various constituents and impurities in the slurry. Slurries which were left without stirring action for up to 24 hours i.e., where the solids had settled to a sediment, were subsequently stirred up to 30 minutes before evaluation. It was observed that these slurries degraded with time in about 8-10 hours due to changes in pH values, viscosity and homogeneity. There were more clumps (10-12%) even after stirring for 30 minutes. Slurries which were continuously stirred did not degrade as fast. For these slurries fibre length distribution changed towards finer particles. Slurries which were allowed to settle and were stirred again degraded much faster than those which were continuously stirred, possibly because of the reactions undergone in the solid form in the sediment. Typical results are given in ***Table 7*** and ***Table 8***.

The foregoing efforts toward slurry development were only a part of extensive work done on it in this phase. The bulk of the efforts were related to studies on slurry homogeneity and flow properties as related to the individual steps comprising the fabrication of insulation felt/tapes as described in the next section.

4.2 Process Development for Ceramic Insulation Felt by the "Slurry -Tape -Casting" Technique

4.2.1 Slurry-Tape-Casting Process

The development of process technology for the fabrication of ceramic insulation felt/tape has taken a significant portion of the over all efforts of the program. Also, the specific objectives and activities have changed to some extent to accommodate changes in the design of the cable from ***Design No.1*** to ***Design No.2***. Earlier efforts to develop extrusion technology which required the use of organic polymer binders were replaced by the "slurry-tape-casting" process technology and most of the efforts have been devoted to this activity. This process technology was developed in two steps. The first step was the development of a "batch" casting system to evaluate design concepts, establish feasibility, and develop operational parameters required for the fabrication of the desired ceramic insulation felt/tape from 20 wt.% BN- 80 wt.% KAOWOOL slurries. Using a system shown in ***Figure 11***, it was experimentally demonstrated that the concept is viable; uniform and homogeneous insulation felt/tapes with widths of 2 inches and lengths up to 2 feet were fabricated. The second step was the development and operation of a manual "semi-continuous" process system. After evaluating several alternative designs based on data obtained in the batch system studies, a design was selected, as shown in ***Figure 12***, for laboratory scale studies and evaluation. In the new design, the felt forming mold was mounted on a movable carriage which traversed on rails. The slurry was fed through a fixed, specially designed nozzle. The quality and the thickness of the felt was controlled by the solids content of the slurry, the rate of flow of the slurry through the nozzle, and the speed of traverse of the mold. A prototype processing facility was constructed with the capacity to fabricate felts/tape with widths of 2 inches and lengths of up to 7 feet. These products were used in fabricating prototype electrical cables for evaluation. As discussed later, the cables met all the high temperature electrical and mechanical property requirements as well as fire burning tests.

The early studies were based on a slurry containing 1 wt.% solids whose material composition comprised 80 wt.% KAOWOOL-20 wt% BN fibers in water. Whereas these experiments proved the correctness of the approach, and had some advantages, they were far from satisfactory for commercial application. The low solids concentration had two specific advantages: the viscosity of the

slurry was low and, therefore, its flow properties were excellent; and the flow of the slurry into the felt making mold through the feed nozzle could be easily controlled. Also, because of the low solids concentration, the material could be uniformly and homogeneously distributed in the felt, a condition necessary for achieving physical homogeneity and uniformity in the insulation felt/tape. A preliminary analysis of manufacturing operations to meet commercial cost requirements indicated that the process should be capable of producing at least 10-20 meters of felt/tape per minute: Production targets could not be achieved economically using 1% solids concentration because of the requirement for large slurry storage tanks, as well as the operational slurry residence requirements in the casting process. These considerations made it imperative to develop slurries with higher solids concentration with the required workability properties in terms of viscosity and settling properties. For example, increasing the solids concentration of the slurry from 1 to 2 wt.% contributes to reduction of the storage tank and factory space requirements by half, and the expected savings are substantially larger if still higher solids concentration in the slurry compatible with operational requirements could be achieved. Hence, the feasibility of using slurries containing 2, 4, 6, 8 and 10 wt.% solids was evaluated.

In addition to solids concentration, the slurry has to meet a number of other property requirements dictated by operational considerations of the "slurry-tape-casting" process. The requirements are: it should be possible to maintain the homogeneity of suspension of solid constituents of the slurry in the tank with appropriate mixing techniques, i.e., the solids should remain in suspension and not settle with time; it should be possible to transport the slurry through a suitable tube to the feeding nozzle without too much viscous drag i.e., using minimum air pressure; and it should be possible to distribute the slurry uniformly through the feeding nozzle into the casting mold. Slurries with a solids content of more than 5 wt.% did not meet these operational requirements. Hence, all further insulation felt/tape development work was carried out with slurries containing 5 wt.% solids.

4.2.2 Development of Automated "Slurry-Tape Casting Process"

In the present phase of the program, the original laboratory "slurry-tape-casting" facility was modified and converted into an automated system: the new design is schematically shown in ***Figure 13***. The automated system was constructed and put into successful operation. Insulation felt/tapes fabricated with this new facility were evaluated for their properties and subsequently used for the fabrication of prototype electrical cables. This new facility was designed to accommodate a scale-up of the process by increasing the slurry holding tank capacity up to 100 liters which enabled continuous fabrication of insulation felt/tape in lengths up to 25-50 feet. A general discussion of some of the important issues governing the scale-up follows:

The preparation of large quantities of a stable slurry and its storage is a difficult task and requires a study and understanding of a number of parameters, specifically: solids content of the slurry; mixing techniques, homogeneity; stability, and flowability: Since these parameters are interrelated, establishing optimum parameters and conditions requires iterative experiments. With increase in solids content, the mixing procedures to attain homogeneity become progressively more difficult. It is necessary to identify and establish appropriate mixing equipment, the rate of mixing, and mixing times taking into consideration the fact that with an increase in mixing speeds and mixing time, the length of the fibers in the slurry tend to decrease which ultimately influences the properties of the insulation felt/tape. Also, with an increase in the solids content of the slurry, its stability decreases, i.e., the solids have a tendency to settle rather than to stay in suspension: This requires establishing the correct pH and viscosity properties of the slurry. Flowability of the slurry, which is also a function of the viscosity, is a critical parameter because it is the key to an automatic slurry-casting process and plays a major role, both in the transfer of the slurry into the casting mold, and in the actual casting process.

The transfer of the slurry from the storage tank into the casting mold is an important step because it controls the quality of the insulation product in the casting process. The viscosity and settling characteristics of the slurry must be such that a uniform flow can be maintained. Since the transfer requires flow of the slurry through a transfer pipe and through a feeding nozzle, it is necessary to study the dynamics of the flow of the slurry, i.e., "viscous drag," through the system in order to establish optimum conditions viz., the length and diameter of the transfer pipe, frictional propertiesf the slurry, the rate of flow as a function of applied pressure. This involves the development of a mathematical model to understand the interplay between the various parameters involved and its use in establishing the process conditions required for obtaining good quality ceramic insulation felt/tape.

Casting the slurry into a tape in the mold is perhaps the most critical step in the whole process. Because of the limitations of the laboratory technique, all of the previous operational experience and experimental data was limited to tape segments with maximum lengths of about 7 feet. Hence, to increase the capability to 25 feet (or more), the following design considerations had to be addressed and optimized: configuration of the slurry-feeding nozzle; casting-mold carriage to accommodate continuous casting; methods of vacuum dewatering, and drying of the insulation felt/tape; and, finally, the mechanism for continuous recovery of the tape.

4.2.3 Mixing and Storage of Slurry

The critical aspects of the slurry making process are the achievement and maintenance of its homogeneity and stability (the length of time it remains in suspension) throughout the volume of the holding tank. A schematic representation of the parameters affecting slurry properties is given in ***Figure 7***. Issues related to homogeneity and stability become much more critical when thick slurries are used. Due to particle interaction, the probability of the formation of clumps of fibers increases rapidly due to the presence of a large number of fiber segments: These clumps can then easily settle giving rise to concentration gradients in the storage tank. Studies and experimental data on the properties of slurries (5% solids of composition 80 wt.% KAOWOOL- 20 wt.% BN fibers) are discussed in a subsequent section.

Although a slurry storage capacity of 500-1000 litres is desirable, a capacity of 100 liters was chosen as a first step after consideration of all the issues associated with higher capacity, particularly mixing, homogenization, and slurry transportation problems. A number of parameters had to be taken into consideration in the selection of the 100 liter slurry tank, particularly: the ability to be used as a hermetically sealed and pressurized vessel; the location and shape of the opening in the top of the container for the insertion of stirring mixer; the location of the inlet tube for the application of pressure to facilitate slurry flow; and the location of the outlet tube for slurry transfer to the casting mold. Several commercially available storage tanks were evaluated/modified using an iterative process to achieve the required optimum capacity and configuration.

Considerable efforts were expended to identify and evaluate suitable impeller blades capable of mixing and keeping the solids in homogeneous suspension; considerations included propeller blade design, mixing speeds and mixing times. These parameters are important because they not only govern homogeneity of mixing but also affect fiber length, i.e., the mechanical action of the blades reduces the fiber lengths. KAOWOOL fibers are short, in the range of 1-3 cm, whereas BN fibers are manufactured by Electrofuel in continuous length of several hundred meters and are cut to the desired length. Earlier laboratory studies indicated the desirability of keeping the BN fiber length in the same range as KAOWOOL fibers in order to minimize mixing time. In the overall mixing process, it is desirable to keep the mixing time to a minimum so as not to reduce the fiber lengths due to mechanical action of the propeller. Several types of commercially available mixers with geometrically different impellers were evaluated for their suitability to achieve homogeneous mixing. It was realized that certain geometries (such as flat vanes) were not conducive to providing significant agitation to a large- volume slurry (about 90 liters) and lead to inhomogeneous mixing. Vanes which extend to the edge of the slurry tank and proprietary geometric shapes to give a vigorous motion to the slurry were

developed in-house. About 20-25 minutes were required to achieve homogeneity with a slurry capacity of 90 litres. Although it is desirable to reduce the mixing time further (to 10 minutes or less), because of time limitations this has not been pursued. Homogeneity was measured by removing small samples at regular intervals and examining tem under a light source.

4.2.4 Fabrication of Insulation Felt/Tape.

From a ceramic felt processing point of view, the flow properties of a slurry are very important for two reasons. The first is that the slurry has to flow smoothly through the system, a fundamental condition to achieve satisfactory operation of the casting machine. Secondly, once it is discharged into the mold, it should flow satisfactorily to achieve physical homogeneity of the felt. In addition to the viscosity of the slurry (governed by solids concentration), the two other important parameters that control the flowability are applied gas pressure and the design of the feeder-nozzle.

The process of fabricating insulation felt/tape from the homogeneous slurry in the holding tank is complex and comprises of a number of critical steps. They are: (a)the flow of slurry from the holding tank via the outlet tube to the slurry feeder-nozzle; (b) design of the feeder-nozzle and flow of slurry to the felt/tape making mold; (c) rate of speed of the casting mold carriage and rate of removal of water; (d) felt thickness control by rollers; and (e) a system for the drying and recovery of tape.

The flow of slurry from the holding tank is achieved by the application of air pressure (compressed air) although for commercial production this could probably be accomplished using electric pumps. This latter option was evaluated but abandoned for the present program for two reasons. The first is that the availability of pumps is very limited in terms of capacity. Although pumps are available for larger capacity use, the smallest capacity pump commercially available was considered to be too large for the small amounts of slurry to be handled in the present study. Moreover, significant efforts had to be devoted to this activity with no previous background and experience. Hence, it was believed to be advisable to adapt and extend the air pressure techniques well established in earlier phases of the program. The slurry flows through a transfer pipe at the bottom of the holding tank into the feeder-nozzle. The rate of flow of slurry depends not only on the applied gas pressure but also on the dimensions of the transfer pipe as well as its material composition. Ideally, a streamline flow is required without significant impedance offered by the wall. To achieve uniform throughput of slurry, the maximum rate of flow should be controlled to avoid turbulent flow through the transfer pipe. Air pressures in the range of 100-200 KPa have been used; the hydrostatic pressure exerted by the 50 cm. high slurry column is less than 3 kPa; which is relatively small and has a negligible influence

on the flow rate. It is necessary to keep the length of the transfer pipe to a minimum consistent with physical dimensional requirements of the system. However, considerable freedom exists in the selection of the diameter. Experiments were performed using 1.25, 1.90, and 2.5 cm diameter Tygon tubing with a length of 30 cm. The slurry flow through these tubes was reasonably uniform. No efforts were made to reduce frictional or other resistance offered by the walls of the tubes. Ideally, slurry flow will be streamlined in the absence of any significant impedance offered by the surface of the wall. Slurry flow rates as a function of air pressure for different diameter transfer pipes are given in ***Figure 14***. As expected, the flow rate is highest at 2.5 cm diameter, and decreases with decrease in diameter to 1.90 cm and 1.25 cm. For any given diameter, however, flow rate is directly proportional to pressure.

The design of the feeder-nozzle is very critical because it not only controls the process speed but also the quality of the felt/tape. Three typical designs, shown in ***Figure 15***, were re-evaluated in terms of: transfer pipe/nozzle interface; nozzle reservoir; and nozzle/felt casting-mold interface. The performance of the third design ***(Figure 15c)*** was considered satisfactory because the feeder comprises two reservoirs: one for receiving slurry and the other for feeding it into the mold thus affording more control. The former is always full and the overflow supplies the second reservoir which feeds the casting mold through a gate. A detailed experimental investigation was carried out to establish the optimum dimensions of the nozzle to achieve satisfactory flow properties for slurries containing 5 wt.% solids.

The design of the casting mold and its associated carriage system was substantially improved so as to be compatible with automatic operation. The length of the casting mold was increased to 12 feet. Improvements to the filter screen were incorporated into the mold to act as a dewatering membrane. An ideal filter should be fine enough to support the slurry with little flow-though, i.e. water cannot pass through the filter without the aid of a vacuum. This is essential for maintaining the slurry in a stable state during feeding into the mold, and for achieving homogeneity in the texture of the felt/tape. The original engineering designs of the system comprising casting mold, as well as the associated movable carriage equipped with dewatering facilities were modified, enlarged and improved to be compatible with the automated system capable of producing a felt/tape with a width of 2 inches and lengths up to 12 feet. The thickness of the felt/tape was controlled by the installation of a system of rollers.

A special felt/tape drying system and a mechanism to collect the dried tape and coil it into a roll was designed, based on concepts shown in ***Figure 16***, and incorporated.

An attempt has been made to optimize the total slurry flow system to improve its performance using a theoretical model. As shown in ***Figure 17,*** the flow of slurry starts from the pressurized reservoir and goes through the transfer pipe, the feeding nozzle, and finally into the tape casting mold. It is clear that the operation of the whole system will be largely dependent upon several variables governed by fluid mechanics principles. The model is based on the following main variables:

- Pressure head due to tank placement, slurry level and air pressure
- Viscosity of the slurry
- Length and diameter of the pipes that transport slurry to nozzle
- Nozzle design and position
- Frictional effects.

The above variables have a direct bearing on the final rate of discharge of the slurry into the casting mold, and, therefore, control the quality of the insulation felt/tape. The following parameters are used in the calculations:

H_1	=	Initial slurry height in the reservoir
P_t	=	Air pressure on slurry
V_p	=	Velocity in the pipe
v	=	Dynamic viscosity
L_p	-	Total length of slurry transfer pipe
D	=	Diameter of pipe
W	=	Specific gravity of slurry

The relationship between the pressure head and discharge is given by the following equation:

$$H_1 + \frac{P_t}{w} = 0.25\frac{V_p^2}{g} + 32v l_p \frac{V_p}{wd^2} + 0.5\frac{V_p^2}{g} \qquad (1)$$

All other variables except the flow velocity can either be measured or constants. Thus equation (1) can be used to calculate the velocity of slurry flow though the pipe.

The volume discharge through the pipe can then be calculated by multiplying the velocity of slurry with cross sectional area. The volume discharge per unit time Q_p is given by

$$Q_p = v_p a_p \qquad (2)$$

where, a_p, is equal to $\pi\frac{d^2}{4}$;

Also, the discharge through the nozzle per unit time, Q_n is given by:

$$Q_n = v_n a_n \qquad (3)$$

where, v_n, is velocity of slurry through the nozzle and a_n is the cross sectional area of the nozzle out let tube. Thus v_n can be computed by:

$$v_n = Q_n / a_n \qquad (4)$$

for the steady state situation Q_n is equal to Q_p.

The volume discharge has to be balanced with the thickness of the felt being made.

The total solids weight required to make a "*l*" length of tape is given by:

$$W_t = l\, b\, h\, p \qquad (5)$$

where b is the width of mold, h, thickness of the felt and p is the density of felt.

Total slurry volume, V_l, required to make "*l*" length of tape is:

$$V_l = W_t / W \qquad (6)$$

which should be equal to the rate of flow of slurry from the nozzle, Qn times the time it takes to move the mold distance "*l*".

If the carriage speed is x meters/sec, then, the rate of deposition of tape is given by:

$$\frac{dl}{dt} = Q_n \frac{t}{bh} \rho \qquad (7)$$

Thus the rate of deposition or mold speed can be calculated from the above equations. It should be noted that the above analysis holds true only for the constant streamlined flow through the nozzle. If for a given experiment the slurry height in the holding tank decreases only marginally, the rate of flow can be assumed to be constant. Alternatively, if p/w is far greater than H_1 in equation (1) then velocity and thus slurry flow will again remain constant. In practice, when a continuous felt making set-up is developed, arrangements will have to be made to keep the term $p/w+H_1$ relatively constant to make felt/tape with uniform properties.

Experimental data showed similar trends to those given by this model. The evaluation and optimization of the "mathematical model" linking the ultimate property requirements of the felt/tape with the process parameters is considered critical for commercial success. It has not been possible to accomplish this in the present program. The ultimate performance requirements of the insulating tape are:

- Physical dimensions (thickness, width)
- Physical properties(density, porosity, microstructure)
- Mechanical properties (tensile and compressive strength)
- Electrical properties(electrical resistivity, dielectric strength)
- Thermal properties(thermal conductivity, thermal stability)

The new casting system has operated satisfactorily. All the process parameters have been optimized and 25-foot felt/tape segments have been fabricated on a routine basis to be used in cable assembly.

4.3 Process Development for Cable Assembly

4.3.1 Cable Assembly

All the prototype cables assembled in this program were fabricated by manual assembly techniques. The procedure comprised of wrapping individual conducting wires with wet insulation felt, binding the two of them together with the polyethylene moisture barrier followed by binding with Cotronic ceramic thread, and drying. Finally, the alumina ceramic protection sleeve was incorporated.

More than 100 prototype cable segments have been fabricated in this program. The cable lengths varied between 2 feet and 10 feet. The length of the prototype cables fabricated was limited by the manual techniques used for their assembly.

Engineering concept studies were carried out to evalaute commercially viable techniques for the assembly of long cables. Two approaches, fundamentally different, were identified. First, the cable assembly can be integrated with the slurry-tape-casting process, i.e., the wet felt from the casting machine is directly fed into the cable assembly process. The second option is to collect the insulation tape in rolls separately, a practice used extensively in the electronic ceramics industry, and use it subsequently as a stock item in the cable assembly process. The advantages and disadvantages of these two approaches for commercial production have not been evaluated in this program.

4.3.2 Electrical Testing and Performance

The electrical performance of high temperature cables is different from that of ordinary cables since their functional requirements are more stringent than being just carriers of electricity. In particular, they are required to operate under fire / high temperature conditions for specific periods of time without electrical failure. The parameters that influence/govern these operational requirements are: electrical resistivity of ceramic insulation felt separating the conducting wires; current carrying capacity of the cable under high temperature conditions; and the burning characteristics of materials used in the fabrication of the cables. Special test facilities were constructed to conduct the required experiments. Prototype cable segments, as developed, have been evaluated throughout the program. The results have demonstrated conclusively that the electrical insulation provided by the 80 wt.% KAOWOOL-20 wt.% BN ceramic felt is adequate to meet all the required specifications; and, that the cable segments meet all the required electrical specifications.

The electrical resistivity of 80% KAOWOOL-20% BN felt was measured as a function of temperature from room temperature to 1000^{o}C, and isothermally at 1000^{o}C for 120 minutes; the results are shown in ***Figure 18.*** Although the values decrease from 10^{13} Ohm-cm at room temperature to about 10^{6} Ohm-cm at 1000^{o}C, the resistivity is still high enough to meet the required specifications.

The *effective current* flowing through cable segments was measured from room temperature to 1000^{o}C. It is defined as a ratio of measured current to the current at room temperature. The experimental data for *effective current* for cable segments with Ni and Ni-clad copper measured using 100 V_{ac} and 4.7 amps current is plotted in ***Figure 19***. It does not change as a function of temperature up to 1000^{o}C. Similarly, as shown in ***Figure 20***, the *effective current* is independent of time up to 120 minutes at 1000^{o}C. These results indicate that both types of electrode materials can be used in practice.

Experimental data for *insulation resistance* of cable segments with Ni and Ni-clad electrode wires as a function of temperature are plotted in ***Figure 21***; the resistance shown is the resistance of cable segment for unit length. The results show that the resistance decreases with increase in temperature from10^{13} ohm.cm at room temperature to 10^{5} ohm.cm at 1000^{o}C. The effect of heating time up to 120 minutes on resistance at 1000^{o}C is shown in ***Figure 22***. Resistance of cable segments decreases for both Ni and Ni-clad electrodes, although at different rates. In any case, the values still remain at about 4 x 10^{3} ohm per cm length of the cable segment and is still capable of providing effective insulation to meet specifications.

4.3.3 Open Flame Burning Tests

Open flame burning tests were carried out using specially designed furnaces, shown in ***Figures 23*** and ***24***, in which the cables could be subjected to open flames using gas-heated burners, capable of operating up to 750°C. The furnaces also permitted concurrent evaluation of cables being developed with other commercially available products under electrically active conditions. Several batches of prototype cable segments (up to 6 feet) have been repeatedly tested and evaluated. The results have demonstrated conclusively that the performance of the cables is satisfactory and meet all the specifications and requirements.

Ceramic felt cables developed in this program, both with and without moisture barrier films, have been evaluated extensively for their performance under open flame burning conditions. The cables did not burn and electrical integrity was maintained for up to 2-3 hours, indicating that they meet the specification requirements listed in Table 1. In contrast, conventional polymer encapsulated commercial cables started to smoke extensively within the first few minutes and electrical breakdown occurred within 15-20 minutes.

A video of the foregoing burn tests was made and has been submitted to DREA.

4.3.4 Moisture Barriers-Need and Status

One of the important requirements for an electrical cable is that its electrical performance should not be adversely affected by exposure to moisture and/or water. In the early stages of the program, using ***Design No.1***, the cable comprised of organic polymer binders in sufficient quantity to provide moisture protection. However, in ***Designs 2*** and ***3***, the polymer binders have been eliminated to decrease /avoid materials which contribute to "fire-load" properties. The ceramic felt insulation used in these cables is porous and can act as a wick to absorb and retain moisture/water. Penetration of moisture through the ceramic insulation can lead to degradation of electrical performance. The presence of moisture can also cause localized arching and subsequent degradation of insulating properties of the ceramic felt layers between the conducting wires. Also, slow corrosion of the metal conductor wires can occur leading to premature electrical failure. Hence, water proofing the cables by the incorporation of a suitable moisture barrier is imperative.

The influence of moisture on the electrical resistance of the insulation felt between the conducting wires in the cable was studied using a specially designed moisture chamber shown in ***Figure 25***. Relative humidity values of up to 90%

could be achieved. Electrical resistance of the cable was monitored using a Solid State Electrometer. Cables with and without polyvinlychloride(PVC) moisture protection film were tested as a function of humidity. The results are shown in ***Figures 26*** and ***27***. As shown in ***Figure 26***, the insulation resistance of the cable without the moisture film protection decreased from 1×10^{12} ohm/cm at 15% RH to 8×10^{7} ohm/cm at 90 % RH, demonstrating that moisture has a deleterious effect on electrical insulation. However, the resistance of the cable with the moisture protection film in place was unchanged indicating that the film is a good moisture barrier.The insulation resistance between the conducting wires in the cable with and without polyvinylchloride film was also measured as a function of time at 85% RH. As shown in ***Figure 27***, the insulation resistance without the film protection decreased rapidly from 1×10^{8} ohm/cm to 1×10^{5} ohm/cm after 50 hours confirming the deleterious effect of moisture on electrical insulation: The insulation resistance of the cable with the film protection remained constant at 1×10^{12} ohm/cm even after 350 hours.

Although use of a PVC moisture barrier was effective, attempts to improve the moisture barrier properties of the insulation felt by incorporating hydrophobic coatings on the KAOWOOL and BN fibres were made to eliminate the problems from terminals (switch boxes, connectors etc.). Attempts to improve the properties using materials such as tetralkyl ammonium silicones, ABS in acetone, polycarbonate in methylene chloride, DOW Corning silicone 92-0009, either by spraying or dipping techniques, were unsuccessful. Hence, a detailed study was initiated to understand the mechanism of moisture migration in the felt. Decyl Agarose was selected based on discussions with its supplier, Sigma Chemical Company. Following the recommendations of the supplier, the material was diluted using 0.5 M HCl. Three different concentrations of 5, 10 and 15 volume per cent were investigated.

Water penetration tests were performed using the following procedure. Ceramic felts of 1 inch wide and 4 inches long were immersed in the hydrophobic solutions for one minute to allow uniform absorption and saturation. The felts were dried in air for at least 12 hours and evaluated using the apparatus shown in ***Figure 28***. Water was filled to a height of 1.5 cm in a one litre beaker. The felt segments, previously treated with the hydrophobic solution were partially immersed in the water for 10 minutes. A series of experiments was conducted to determine the level of penetration of water in the felt. The level was determined by observing the color change in the felt. A typical example for the effect of treatment with 10% solution is shown in ***Figure 29***. Normally, untreated felts should show a rapid increase in water penetration due to capillary action. Within 3-4 minutes, the water rises to a height of 7 cm and further penetration is slow. Contrary to expectation, the hydrophobically treated material treated test sample showed higher penetration rates than untreated samples. Experimental results showed that the water penetration in the treated samples was

slightly faster and reached slightly higher level (7.5 cm). One possible explanation for water reaching a higher level in the treated felts is that treatment with hydrophobic material leads to a narrowing of the pore openings in the felt. According to theory, the size of a capillary opening is related to the height of water column by the equation:

$$h = 0.046 / D$$

where, h is the height of the water level and D is the diameter of the capillary. Using the above equation, the average diameter of the capillary is calculated to be 66 microns for untreated felts and 61 microns for treated felts.

The diameters of the capillaries were calculated using two other approaches. One such approach utilized Secondary Electron Micrograph of the ceramic felt as shown in ***Figure 30***. Distribution of voids was utilized to determine the average size of voids in any given plane. The area of all visible cavities was determined and was used to calculate diameter of an equivalent surface area circle. From this data, the average diameter of the pores was calculated to be 25 + microns.

The third approach is based on using the density of felt, and the theoretical density and diameter of KAOWOOl and BN fibres. For this first order calculation, the following assumptions were made:

- The fibres are uniformly distributed in three dimensions
- The average pore size is the same
- The average length of the fibres is 0.25 cm and is arranged parallel and in an orderly manner.

The following parameters were used in the calculations:

- density of the felt: D_f + 0.41 g/cm^3
- theoretical density(composite density): D_c = 2.369 gm/cm^3
- average diameter o fibres: D = 5 microns
- thickness of felt : t = 0.25 cm

For the purpose of these calculations, a felt in the form of a cube with unit length of 0.25 cm is assumed. The volume of this felt is given by the expression:

$$V_1 = 0.25 \times 0.25 \times 0.25 \text{ cm}^3 = 1.5625 \times 10^{-2} \text{ cm}^3$$

The weight of the sample felt is:

$$W_1 = V_1 D_f = 1.5625\ 10^{-2}\ cm^3 \times 0.41\ g/cm^3 = 6.4 \times 10^{-3}\ gm$$

The weight of each fibre of length 0.25 cm:

$$W2 = D^2 L D_c / 4 = 3.14 \times 5\ cm \times 5\ cm \times 0.25\ cm \times 2.369\ g/cm^3 \times 10^{-9}/4$$

$$= 1.1625 \times 10^{-7}\ gm$$

The number of fibres in the sample felt is:

$$N_1 = W_1 / W_2 = 0.1025/1.1625 \times 10^{-7} = 5.5 \times 10^4$$

Supposing that the fibre distribution in the felt is three dimensional and has equal number of fibres in each dimension, the number of fibre, X, in each dimension is given by:

$$X = 3 \quad N_1 = 38$$

Since the thickness of each fibre is 5 microns, then 38 fibres will occupy 190 microns of space. Thus the total void length is:

$$D_2 = 2500 \text{ micron (felt width)} - 190 \text{ micron} = 2310 \text{ micron}$$

Therefore, pore size in each dimension in the first order approximation is:

$$A = D_2 / X = 2310 / 38 = 61 \text{ micron}$$

Thus, the average pore sizes from three different approaches are:

Capillary theory	61 microns
SEM	25 microns
Calculated	61 microns

The most realistic number is probably from the SEM evaluation. The calculated numbers using the capillary theory and volumetric considerations are also in the same order of magnitude.

The results of the hydrophobic material studies suggest that the problem of preventing moisture penetration from terminals (switches, junction boxes etc.)

cannot be achieved by treating felts with hydrophobic materials, and this is not a viable approach.

In the period of this report considerable research efforts were devoted to analyze the moisture/water problem from a design point of view, i.e., whether opportunities existed to solve the problems by design modifications. Since the origin of the problem is associated with the porosity of the felt, one possible solution is to replace it with another more suitable material. This was considered impracticable for a number of reasons. Firstly, the materials comprising the ceramic felt, by themselves, are satisfactory both in terms of their chemical stability and electrical property requirements and are not directly contributing to the problem. Hence, replacing these materials is not the answer. Secondly, moisture/water problems can be completely eliminated by the use of these same materials in the form of monolithic ceramics: however, this compromises the flexibility of the cable and therefore is not an option. The only viable option appears to be to reduce the porosity of the felt. This approach involves studying the relationship between the porosity of the felt and the moisture/water performance of the cable. Although there is some merit in this approach, practical considerations rule it out because it involves increased effort in terms of time and R&D cost; and, because, there is no guarantee that a satisfactory solution can be found.

Considerable efforts were spent in identifying commercially available polyethylene based films and evaluating them for moisture barrier applications according to ***Cable Design No3***. The results have been satisfying. By keeping the weight of the films to a minimum, the *"fire load"* on the cable can be kept to a minimum: Test measurements indicate that the weight of the polymer film is less than 0.1-0.2 wt.%. This approach offers several other advantages: the polymer film is very inexpensive; and it can be incorporated in the cable manufacturing process not only as a moisture barrier but also as a means of handling the ceramic felt in the casting process. Experimental data accumulated to date on the performance of the cables during burning tests indicate that the use of a polyethylene moisture barrier does not contribute to fire load. In conclusion, it is felt that the foregoing approach is the best, given the status of technology. The problems in areas where wires are joined, e.g., switches, junction boxes etc., need to be solved separately.

5.0 SUMMARY AND CONCLUSIONS

1. The overall objective of the program, i.e., to develop a fire-safe electrical cable stable to temperatures of up to 1000 °C for at least one hour, has been successfully accomplished. The individual steps contributing to this success are summarized below.

2. Using an iterative process, a satisfactory engineering design and a processing technology suitable to meet the material needs have been developed for the fabrication of the cable. The design is based on the use of ceramic insulation felt comprising 80 wt.% KAOWOOl-20 wt.% BN fibres, with polyethylene as moisture barrier, and Ni-clad copper as electrically conducting wires.

3. A commercially viable "slurry-tape-casting" process has been successfully developed for the fabrication of insulation felt/tapes. The insulation has been tested, and meets all the electrical and physical flexibility property requirements for use in cables.

4. Prototype cables with lengths of up to 12 feet have been successfully fabricated and tested. They meet all the required specifications. They have operated at temperatures up to 1000 °C for at least one hour without electrical failure. They also withstand open flame burning for more than 3 hours.

Table 1. Property Requirements for Fire-safe Cables

PROPERTY	COMMENTS
FIRE PERFORMANCE	Wire must operate in the presence of a Class A (1000 C) fire or hotter (propellant fire is 1600 C)
FUEL LOAD	Total combustible material should be as low as possible
FIRE SPREAD	Flame spread should be minimal in order to reduce spread of fire through wire ducts
SMOKE TOXICITY	Smoke should not be acutely toxic. This precludes use of HF- and HCl- producing materials containing halogens, cyanide-producing organics, etc.
NORMAL ELECTRICAL PERFORMANCE	The wire must perform excellently in normal non-fire circumstances
WATER RESISTANCE	The wire should neither wick nor absorb water
WEIGHT	The wire must be lightweight since when used in substantial quantities it becomes a significant portion of total topside weight
USER CONSIDERATIONS	The wire must flex, strip and handle like existing electrical wire Connection capability is required
COST	The cost of this wire should not greatly exceed that of the current standard wire

Table 2. Properties of BN Fibers

Fibre diameter (μm)	3--6
Composition	92 wt.% BN 7 wt.% B_2O_3 1 wt.% other
Density (g/cm^3)	1.6--2.0
Tensile strength (GPa)	0.8
Modulus (GPa)	10
Crystal structure	Turbostatic (below 2000°C) Hexagonal (over 2200 °C)
Melting point (°C)	3000
Maximum usable temperature (°C): I. or R. A *	2500
O. A **	1000
Thermal conductivity (W/mK)	
at 20 °C	70
500 °C	50
1000 °C	35
Dielectric constant (f= 8.5 GHz)	4.0
Resistivity (Ω-cm)	$--10^{13}$ at R.T. $--10^{8}$ at 1000 °C
Dielectric strength (kV/mm)	30.0

* I. or R.A: Inert or Reduced Atmosphere
** O. A: Oxide Atmosphere

Table 3. Sources and Compositions of Alumino-silicate Fibers

Material Tradename	Composition (%) Al_2O_3	SiO_2	Na_2O	Fe_2O_3	CaO	MgO	Manufacturer/ Supplier
FIBREFRAX $13.33/lb	51.9	47.9	0.08	0.1			Carborundum/ Williams & Wilson
FIBREMAX $34.25/lb	72.0	27.0	0.1	0.02	0.05	0.05	Carborundum/ Williams & Wilson
KAOWOOL $3.70/lb	47	53		0.15			Thermal Ceramics

Table 4. pH and Viscosity Properties of Water Slurries Containing FIBERFAX, FIBREMAX, and KAOWOOL.

Compositions and Properties of FIBREFRAX Water Slurries

Wt. % Water	Wt. % Fibres	Mixing Time* (min)	pH	Viscosity** (CPS)	Comments
97.5	2.5	5	5.8	302	- Blender capacity is reached at 10% solids.
95	5	5	5.5	387	- Workable slurry is at 5% solids.
92.5	7.5	10	5.6	448	
90	10	20	5.4	520	

Compositions and Properties of FIBREMAX Water Slurries

Wt. % Water	Wt. % Fibres	Mixing Time* (min)	pH	Viscosity** (CPS)	Comments
90	10	5	6.4	65	- Blender capacity is reached at 35% solids.
85	15	10	6.4	88	- Workable slurry is at 25% solids.
80	20	15	6.4	118	
75	25	20	6.4	194	
70	30	30	6.3	240	
65	35	40	6.2	341	

Compositions and Properties of KAOWOOL Water Slurries

Wt. % Water	Wt. % Fibres	Mixing Time* (min)	pH	Viscosity** (CPS)	Comments
95	5	5	7.7	124	- Blender capacity is reached at 20% solids.
90	10	10	7.7	140	- Workable slurry is at 10% solids.
85	15	15	8.1	680	
80	20	30	8.4	1320	

* Mixed in Osterizer 8 blender.
** Shear rate of 30 RPM.

Table 5. Settling Time and Viscosity of 80 wt.% KAOWOOL-20 wt.% BN Fiber Sluries as a Function of Solids Content

Number	Composition wt.%	Viscosity cps	Settling Time Minutes	Remarks
1	0	10		pure distilled water
2	2	80	5	
3	4	165	3.5	
4	6	550	2	
5	8	1500	not clear	thick slurry
6	10	2500	not clear	thick slurry

• Viscometer: Bohlin Visco 88

•• Distilled water is used for all slurries

••• pH is adjusted at 7.0

Table 6. Slurry Characteristics for 5 wt.% Solids Content

Property	Values
Viscosity	220 -245 cps
pH	6.8 - 7
Settling Time	2.7-2.8 minutes
Homogeneity	Clear with less than 1% clumps
Fibre Length Distribution	1 - 4 cm

Table 7. Stability vs Time Experiments for 5 wt.% Slurries (not stirred)

Time hours	Viscosity cps	pH	Homogeneity	Settling Time minutes
1	238	7.0	< 1% clumps	2.8
2	235	7.0	< 1% clumps	2.8
4	235	6.9	3% clumps	2.6
6	238	6.9	4% clumps	2.3
8	252	6.8	8% clumps	1.9
10	262	6.8	10% clumps	1.7
18	290	6.7	10 % clumps	1.6
24	296	6.6	12% clumps	1.5

Table 8. Stability vs Time Experiments for 5 wt.% Slurries

Time hours	Viscosity cps	pH	Homogeneity	Settling Time minutes
1	235	7.0	<1% clumps	2.7
2	235	6.9	<1% clumps	2.7
4	232	6.9	<1% clumps	3.0
6	225	6.7	<1% clumps	3.1
8	222	6.6	<1% clumps	3.2
10	215	6.7	<1% clumps	3.3
18	212	6.6	<1% clumps	3.8
24	198	6.6	<1% clumps	4.2

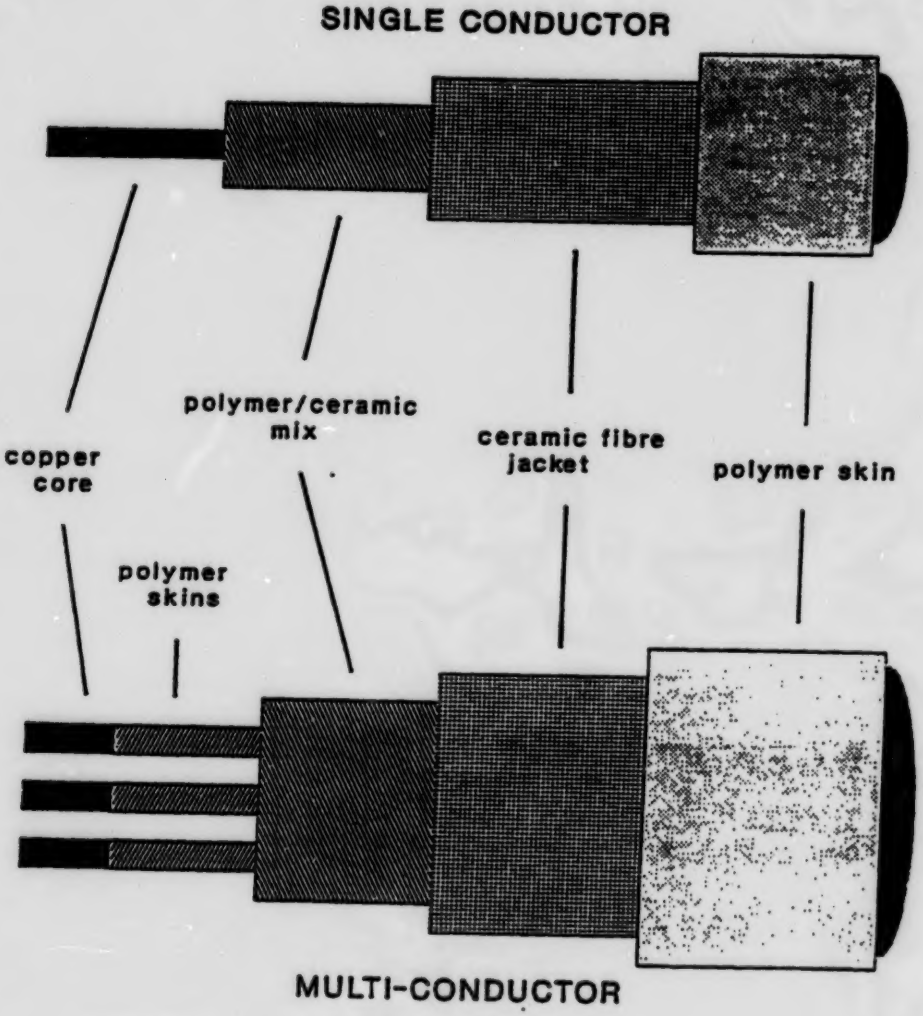


Figure 1. ***Design No.1*** **Suitable for Extrusion Technology**

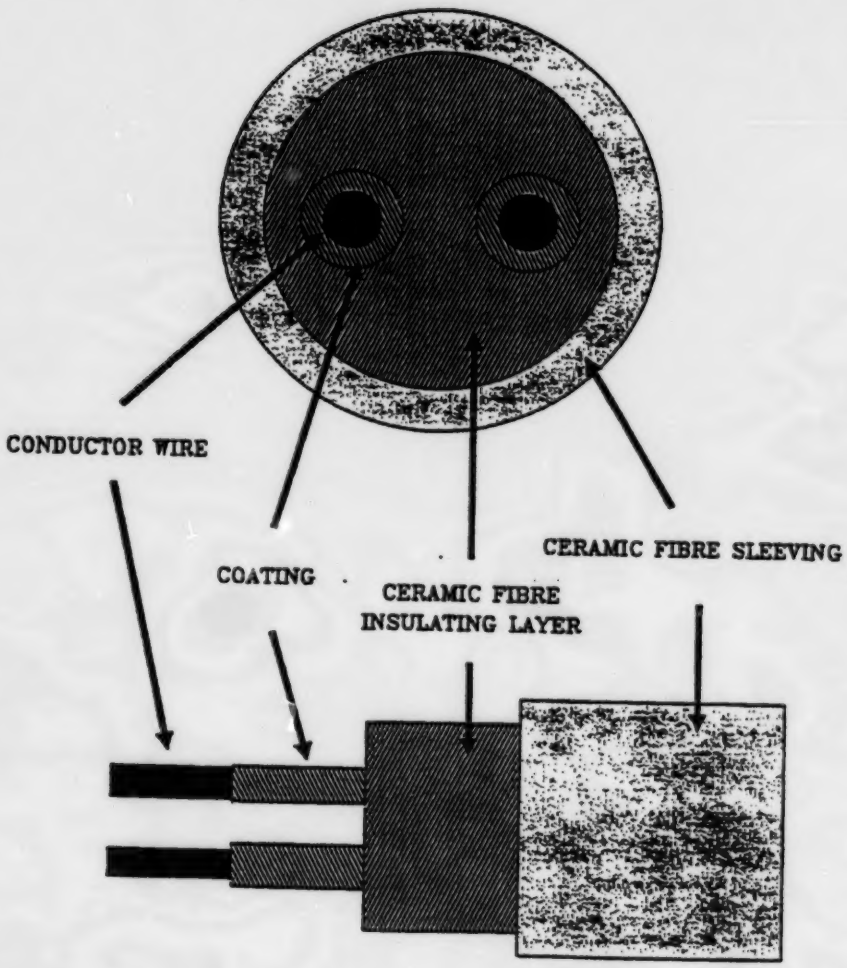


Figure 2. ***Design No.2*** **Suitable for Slurry-Tape-Casting Technology**

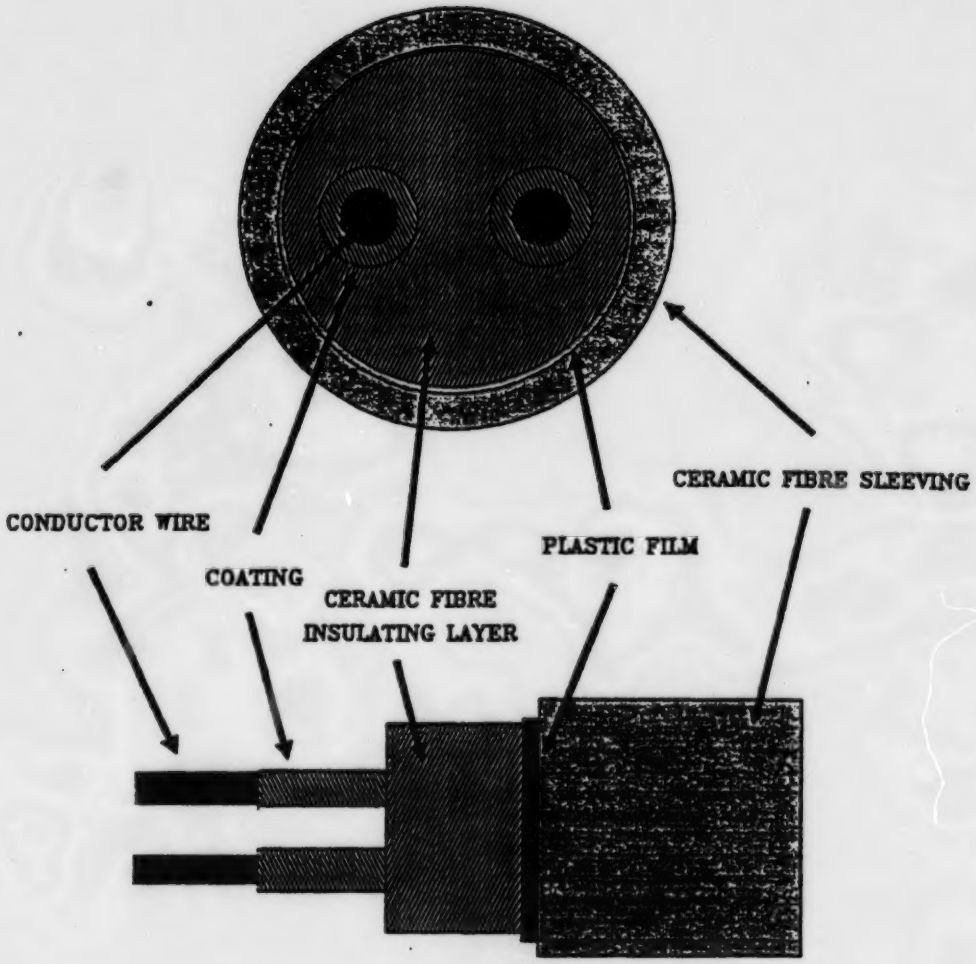


Figure 3. ***Design No.3*** **Suitable for Slurry-Tape-Casting Technology (Modified to include Moisture Barrrier)**

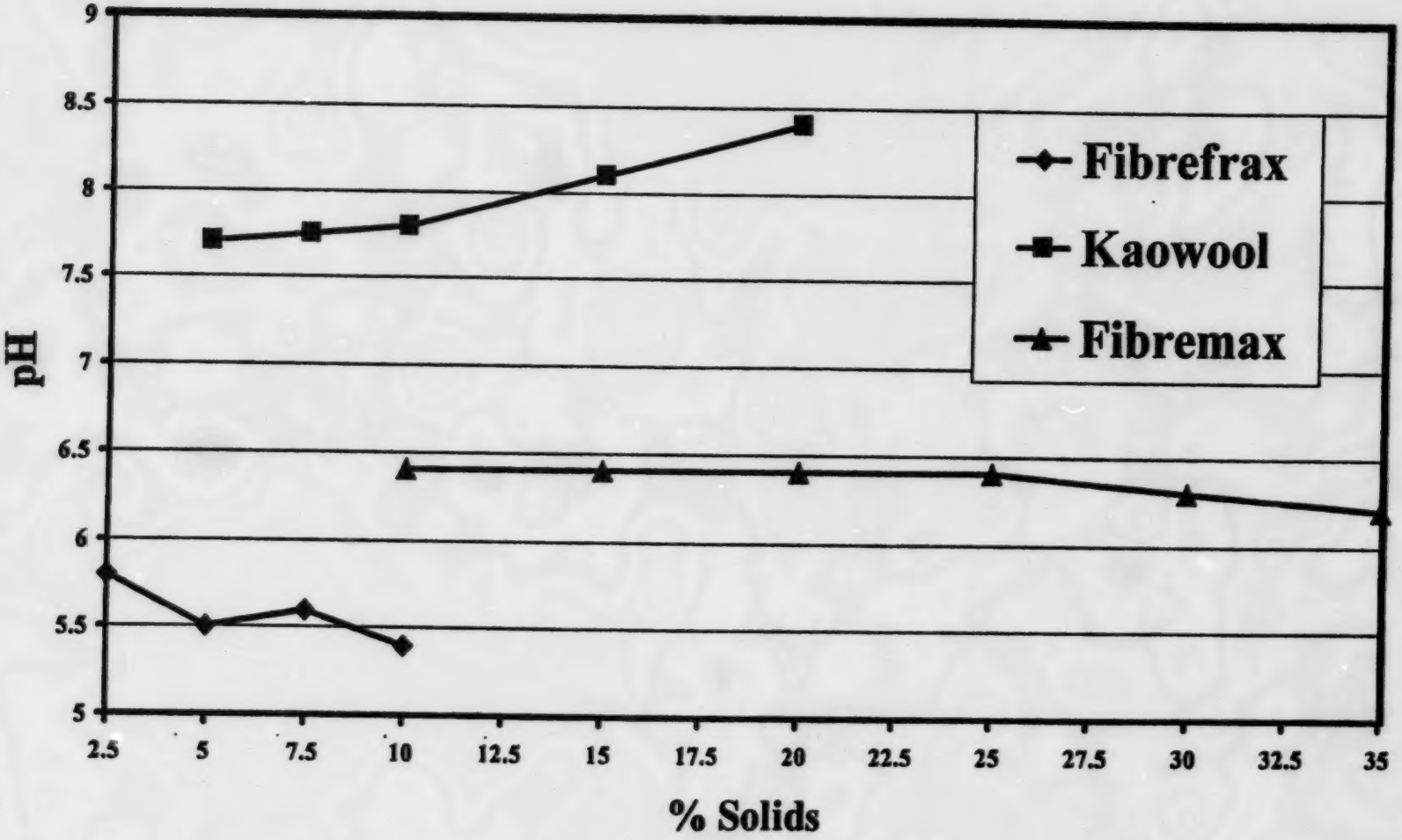


Figure 4. pH as a Function of Solids Content for FIBREFAX, FIBREMAX, and KAOWOOL Sluries

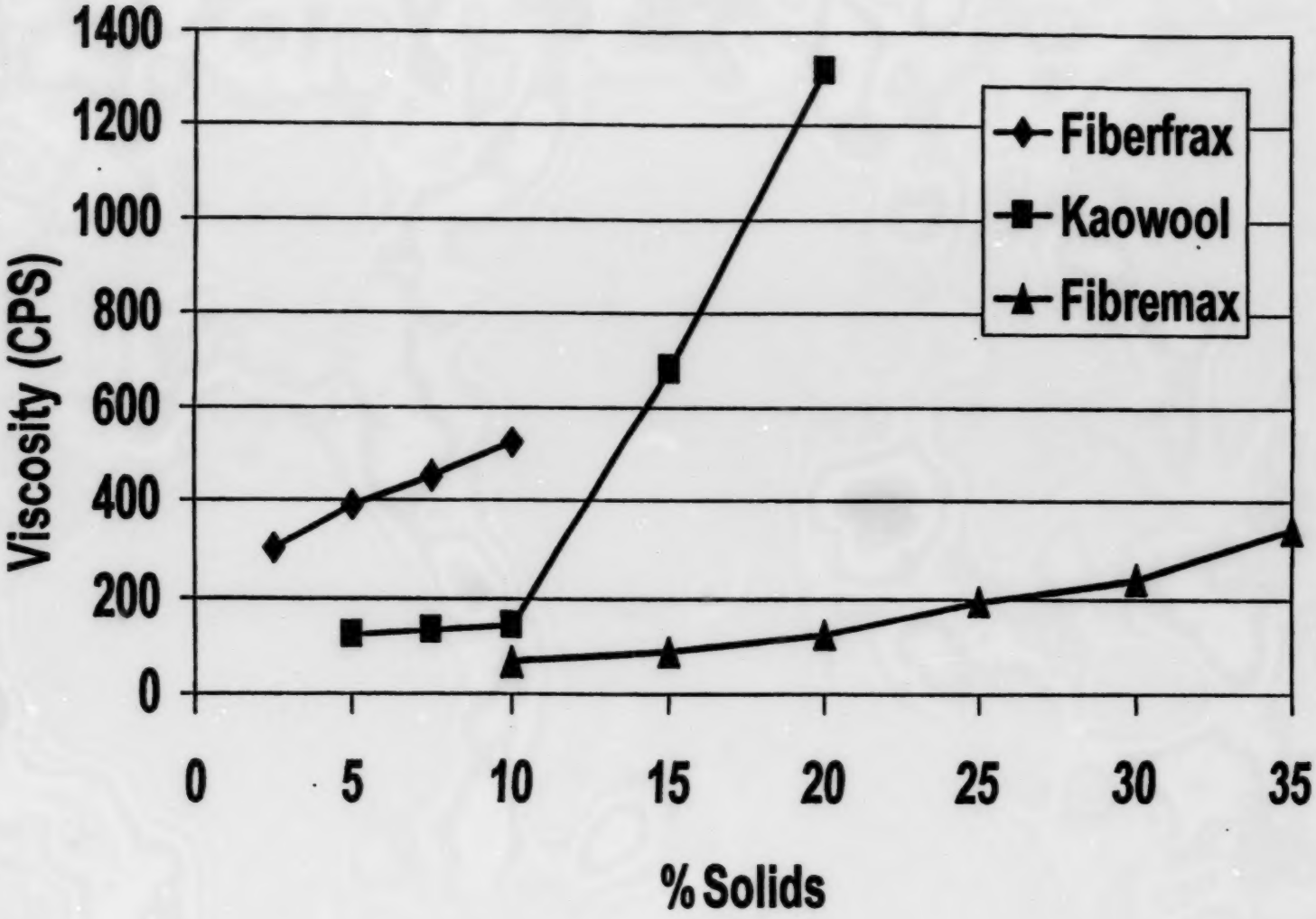


Figure 5. Viscosity as a Function of Solids Content for FIBREFAX, FIBREMAX, AND KAOWOOL Slurries

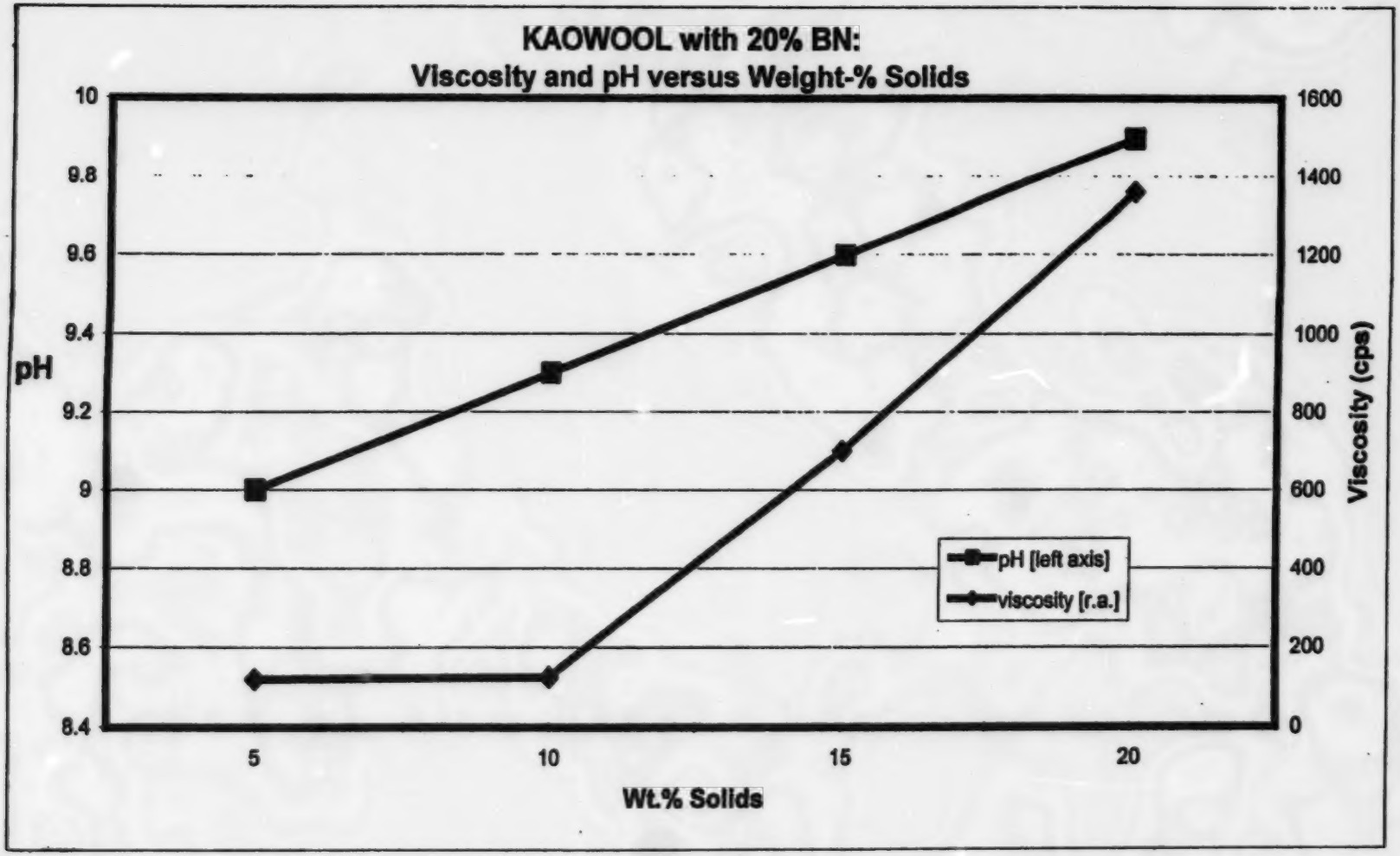


Figure 6. pH and Viscosity as a Function of Solids Content for 80 wt.% KAOWOOL - 20 Wt.% BN Fiber Slurries.

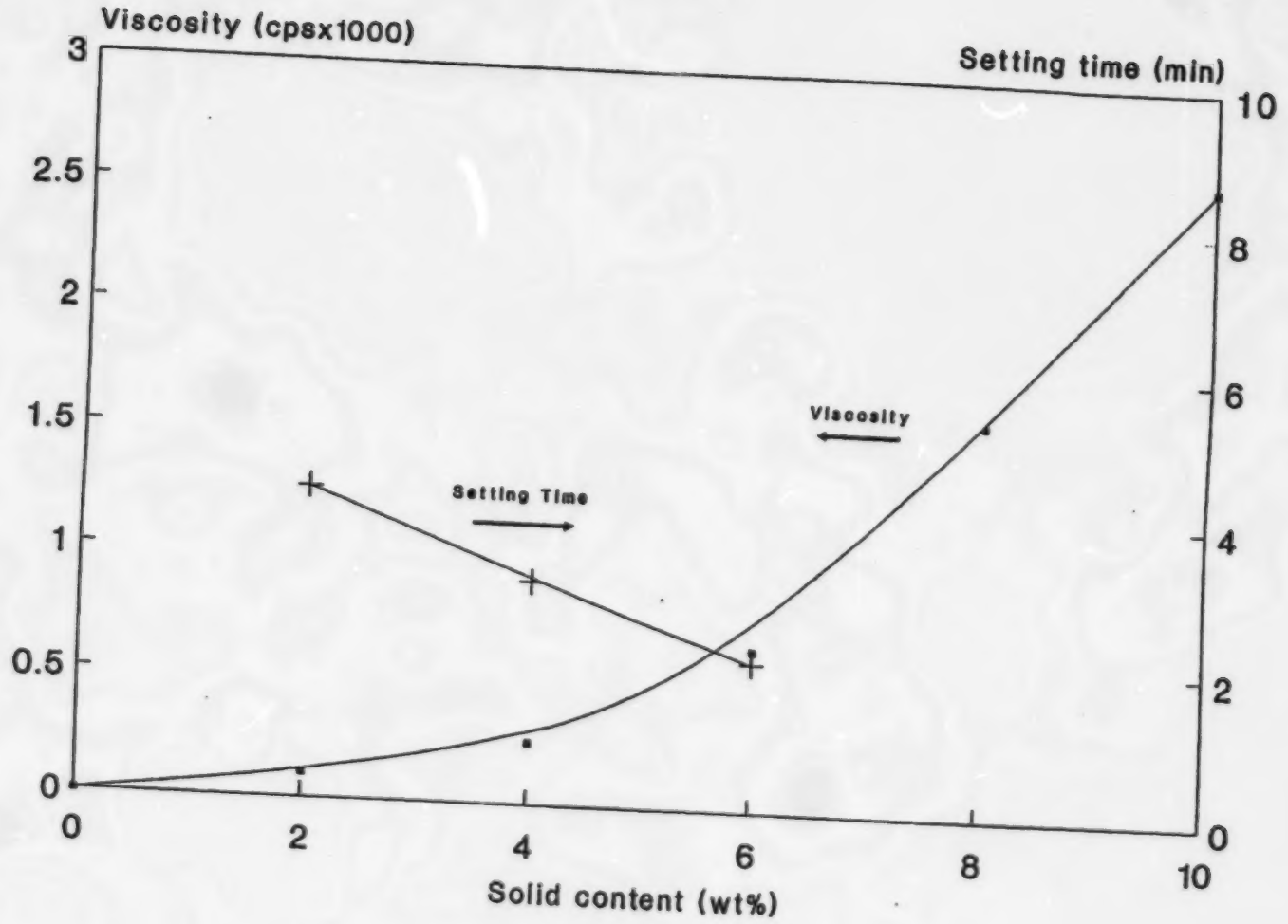


Figure 7. Settling Time and Viscosity for 80 wt.% KAOWOOL-20 wt.% BN Fibre Slurries as Function of Solids Content.

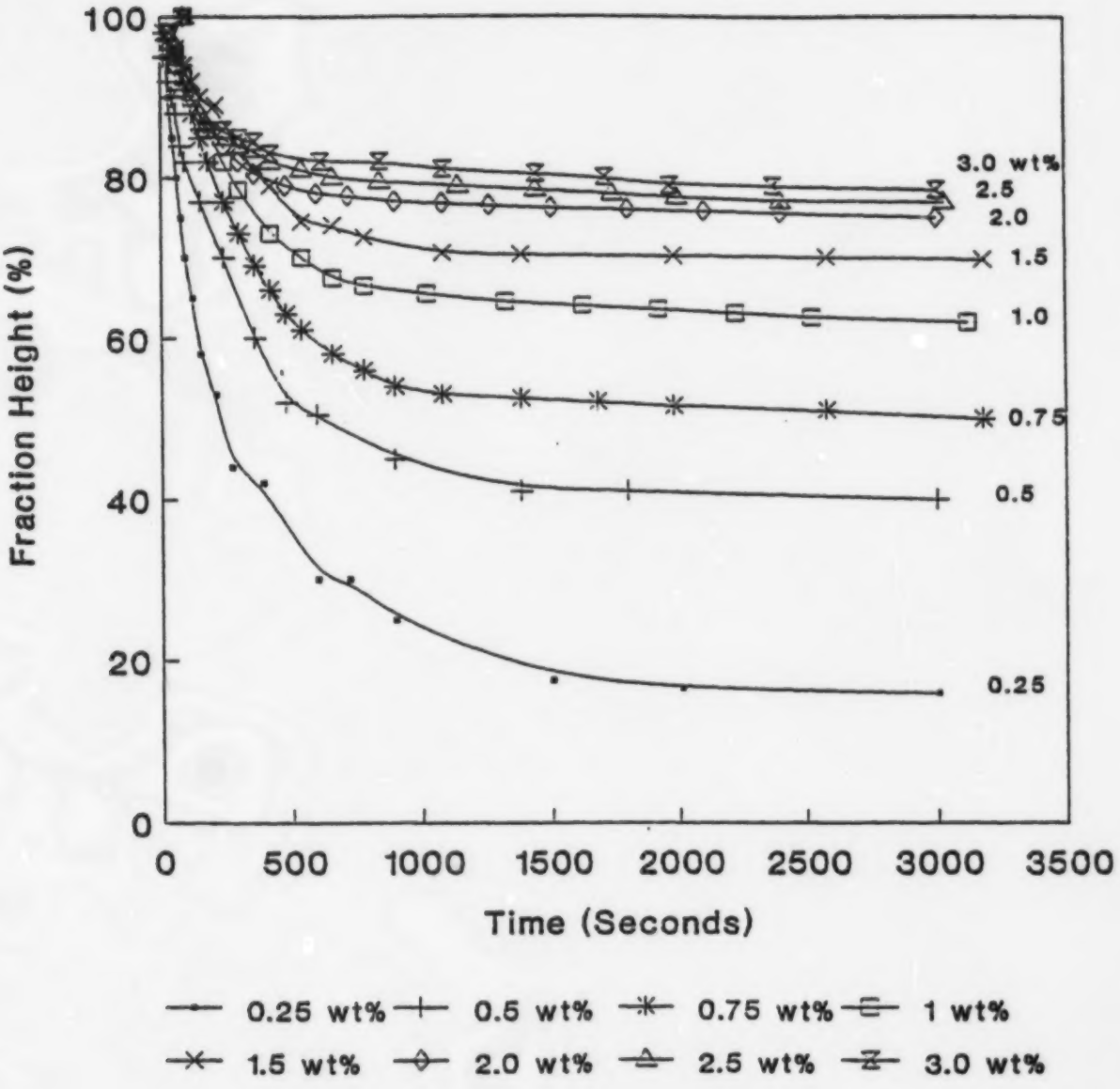


Figure 8. Slurry Height as a Function of Time for 80 wt.% KAOWOOL-20 wt.% BN Slurry

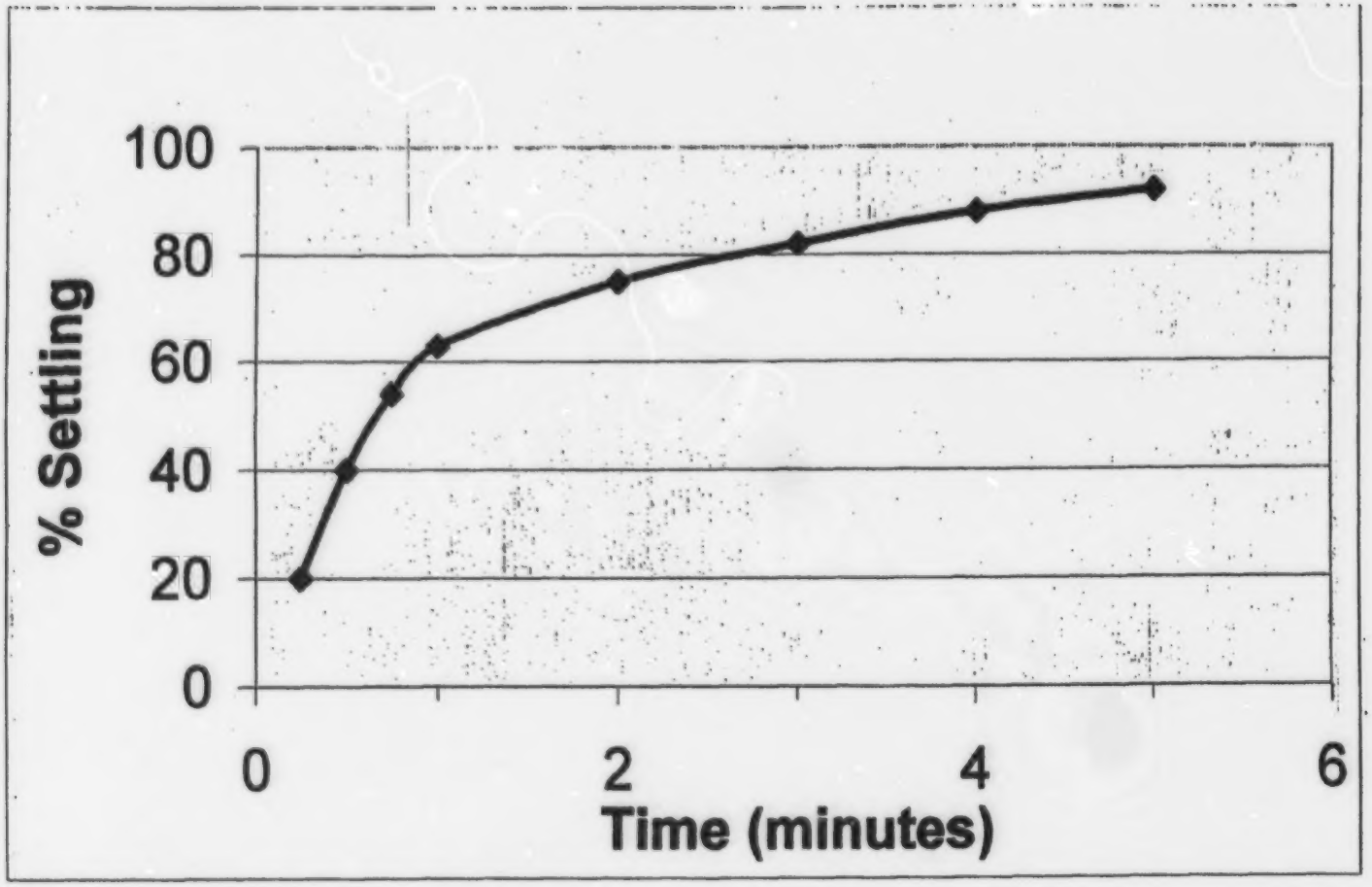


Figure No. 9: Projection of Settling Characteristics for 5 wt. % Solids Slurry

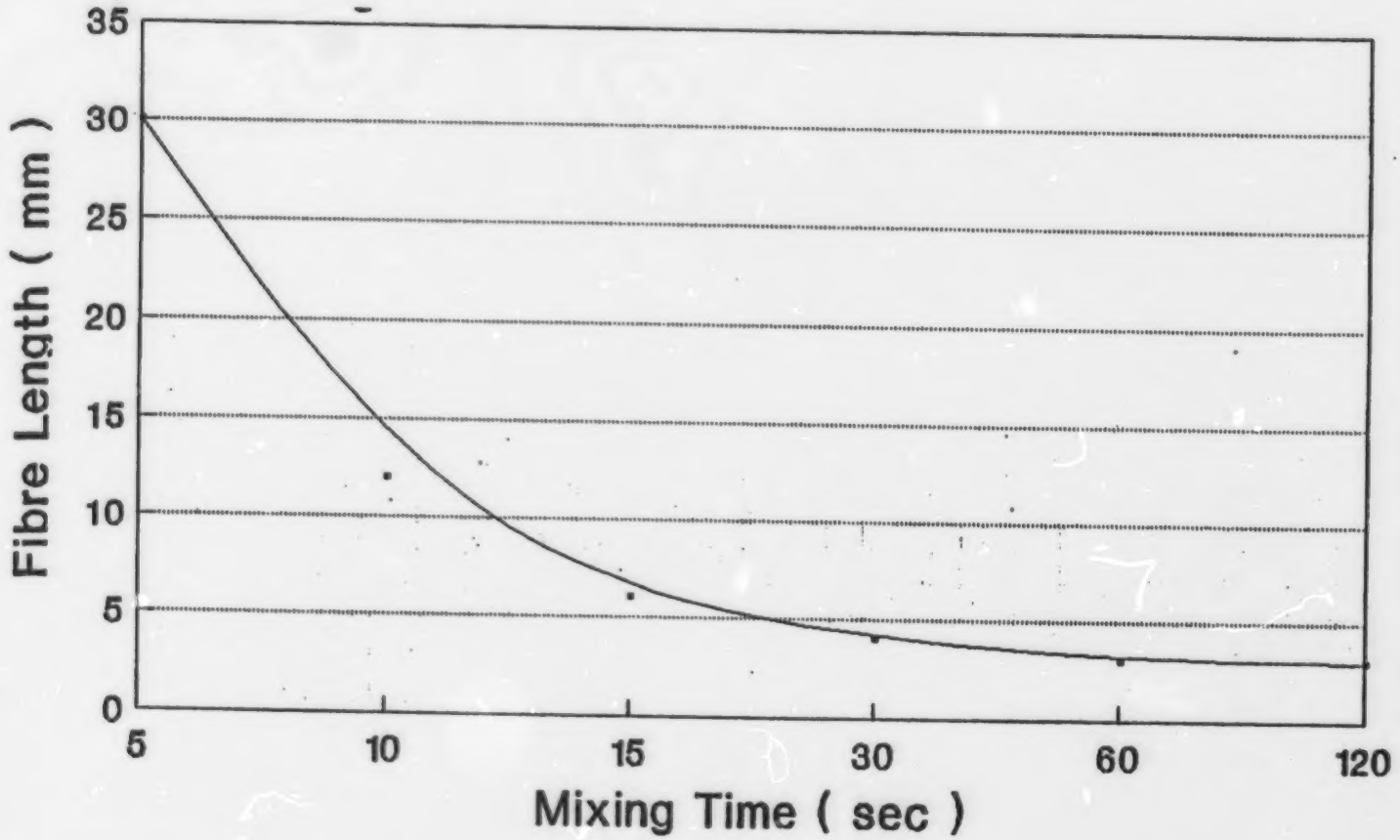


Figure 10. Decrease in BN Fiber Length as a Function of Mixing Time

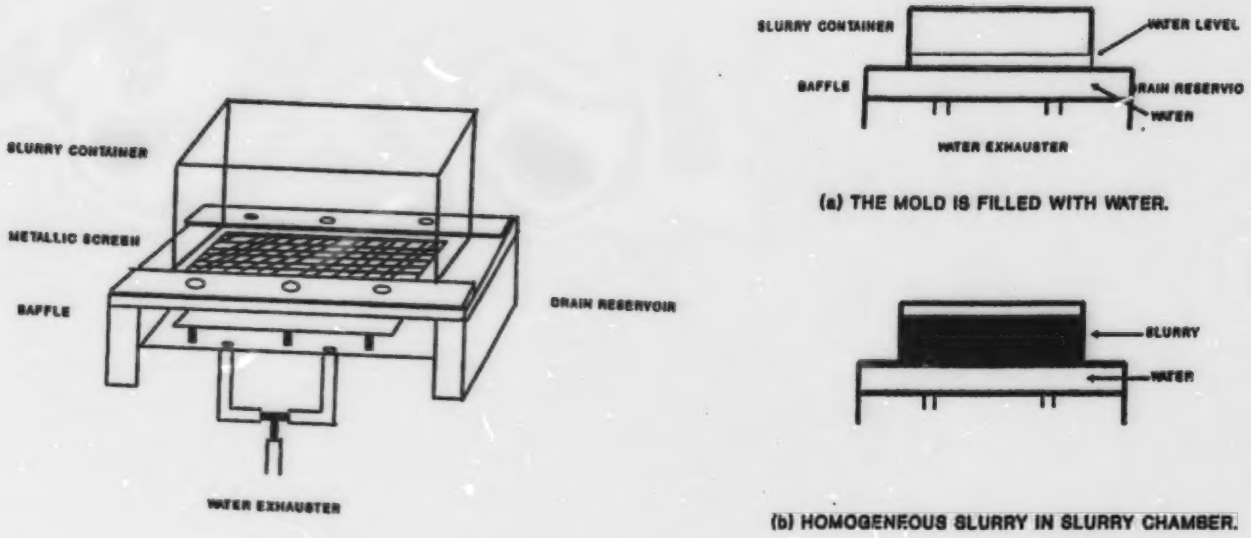


Figure 11. Batch Process for Making Insulation Felt

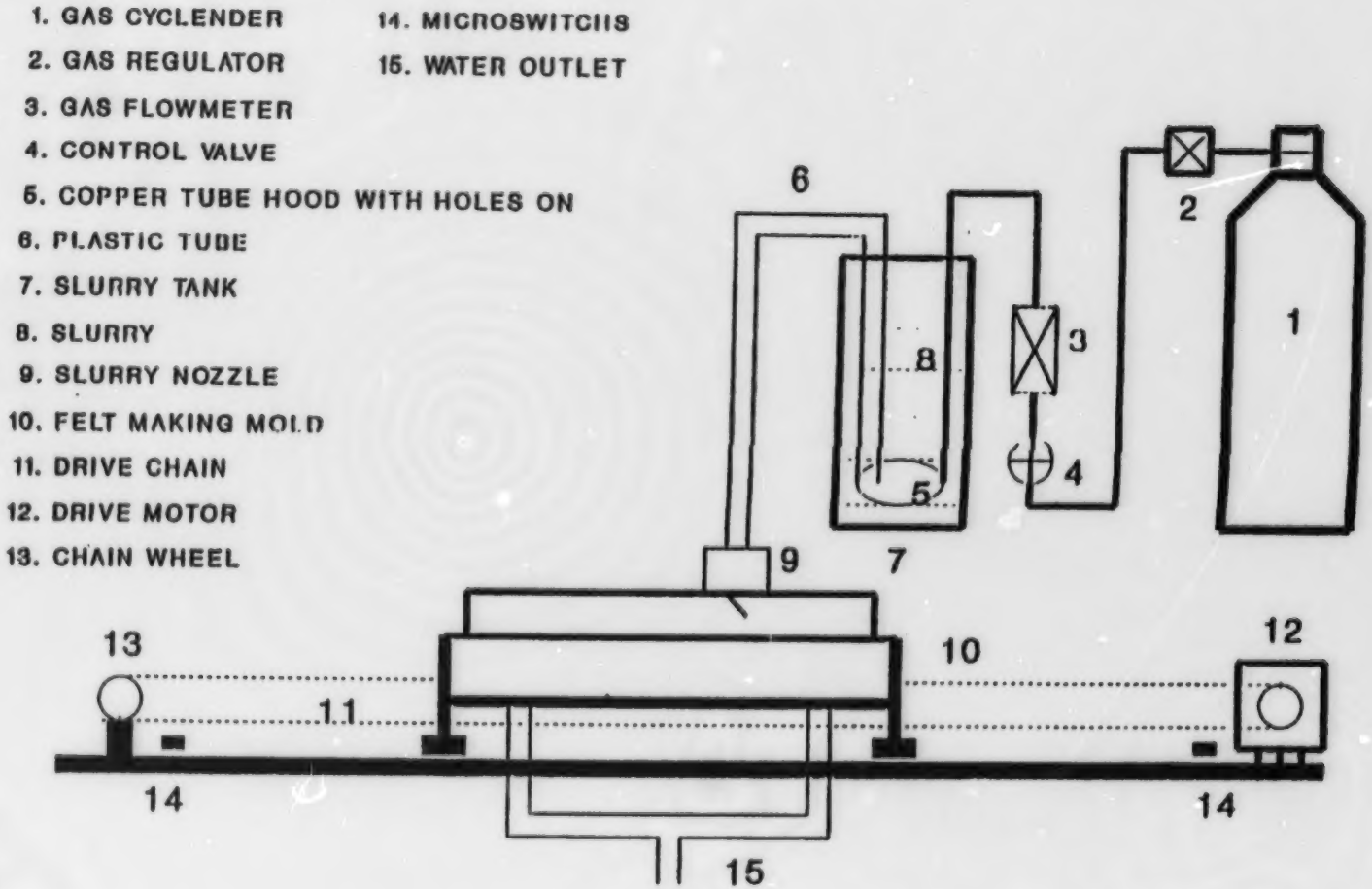


Figure 12. Semi-Automatic Slurry-Tape-Casting Process

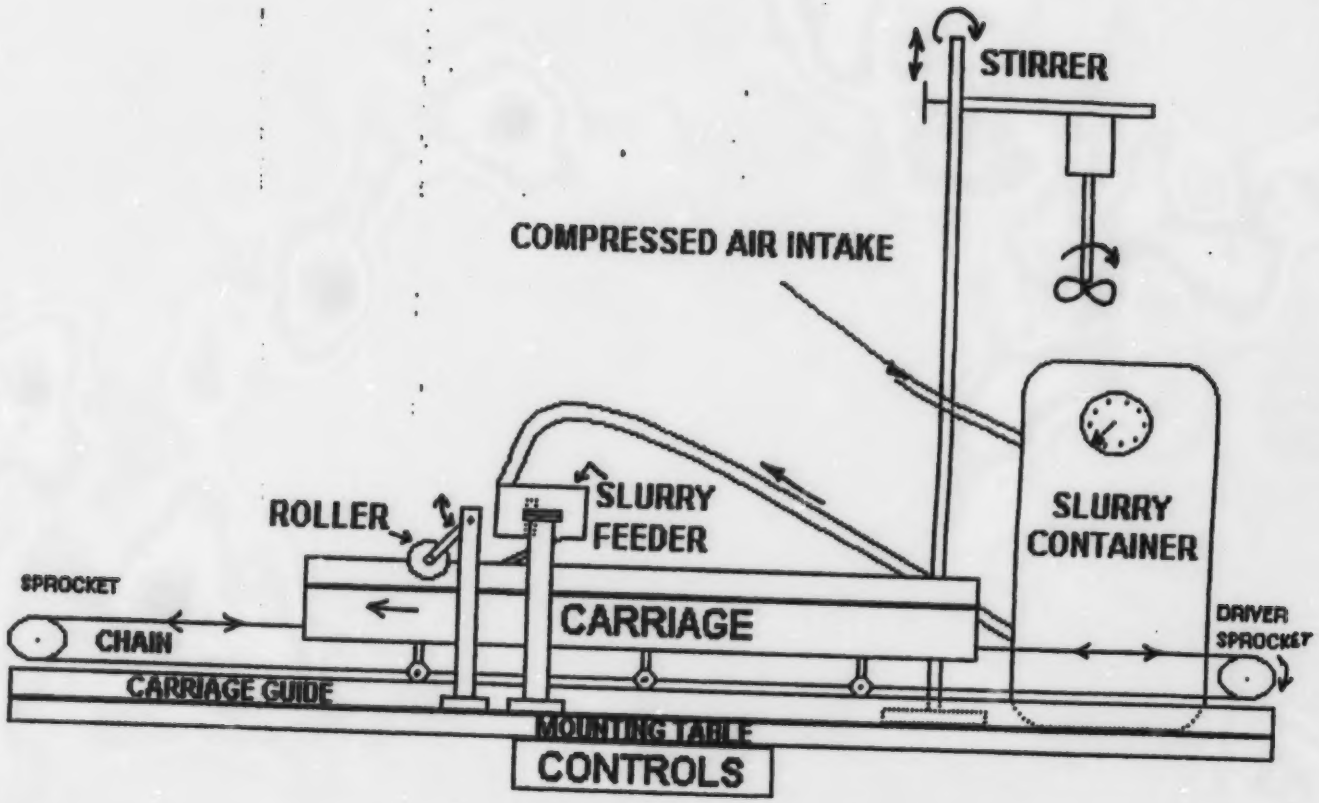


Figure 13. Automatic Slurry-Tape-Casting Process

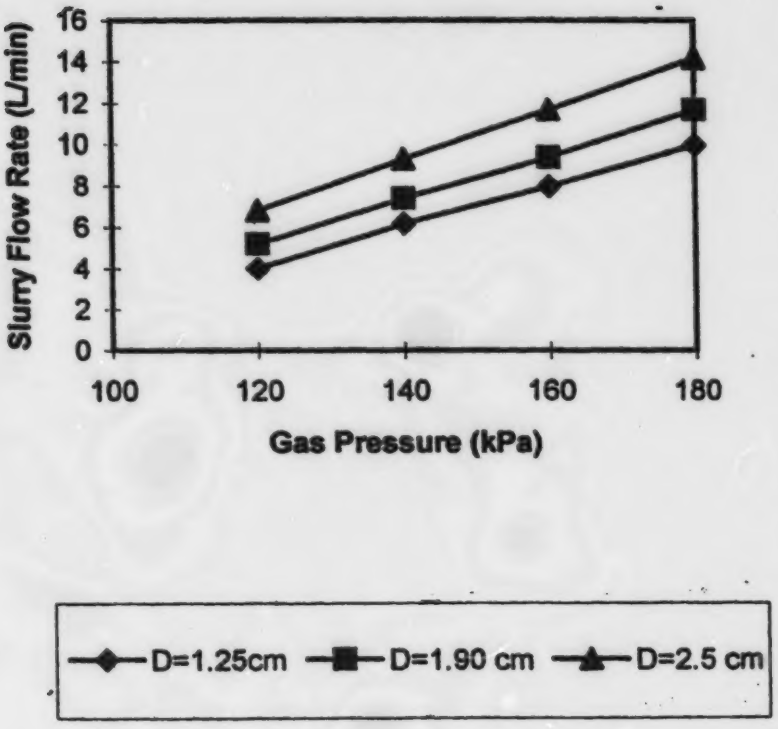


Figure 14. Slurry Flow rate as a Function of Diameter and Air Pressure

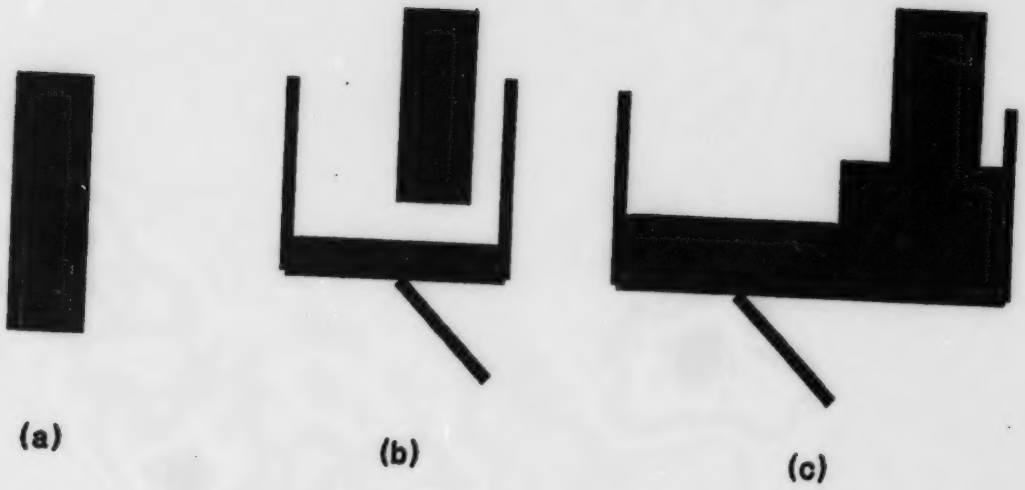


Figure 15. Types of Feed Nozzle Designs Evaluatd

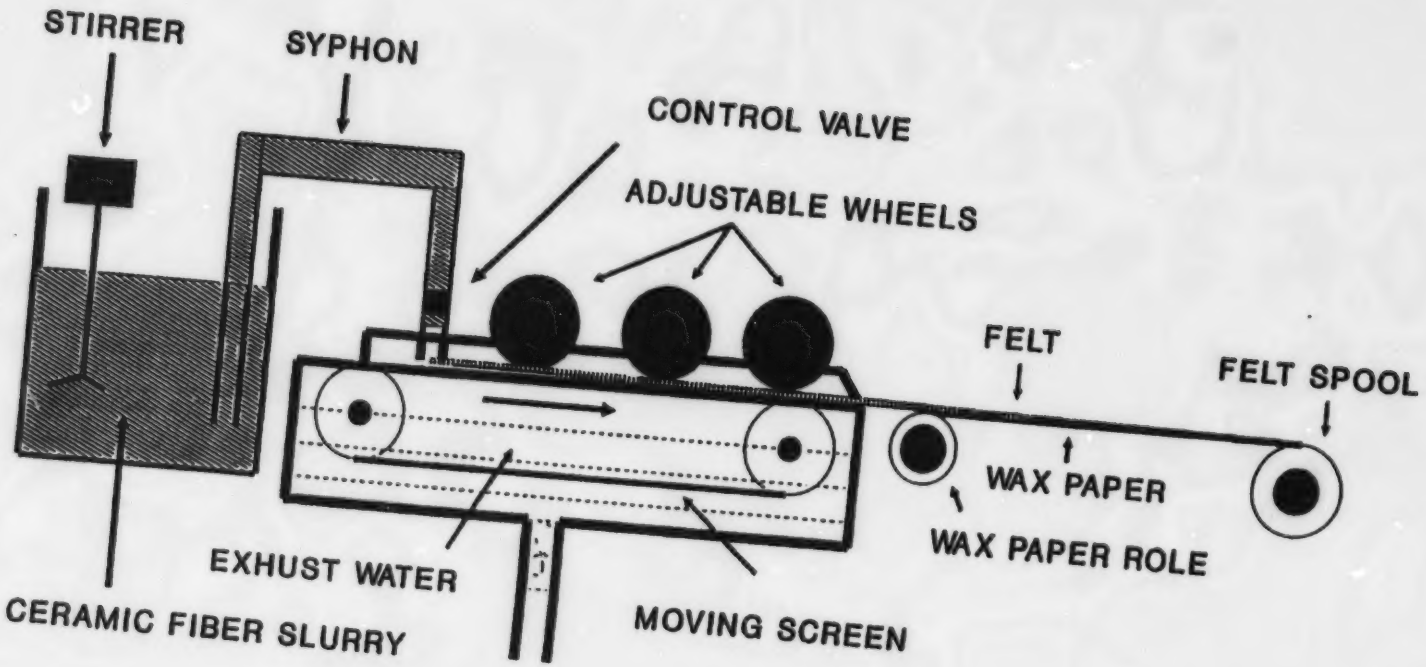


Figure 16. Concept for Continuous Recovery of Insulation Tape

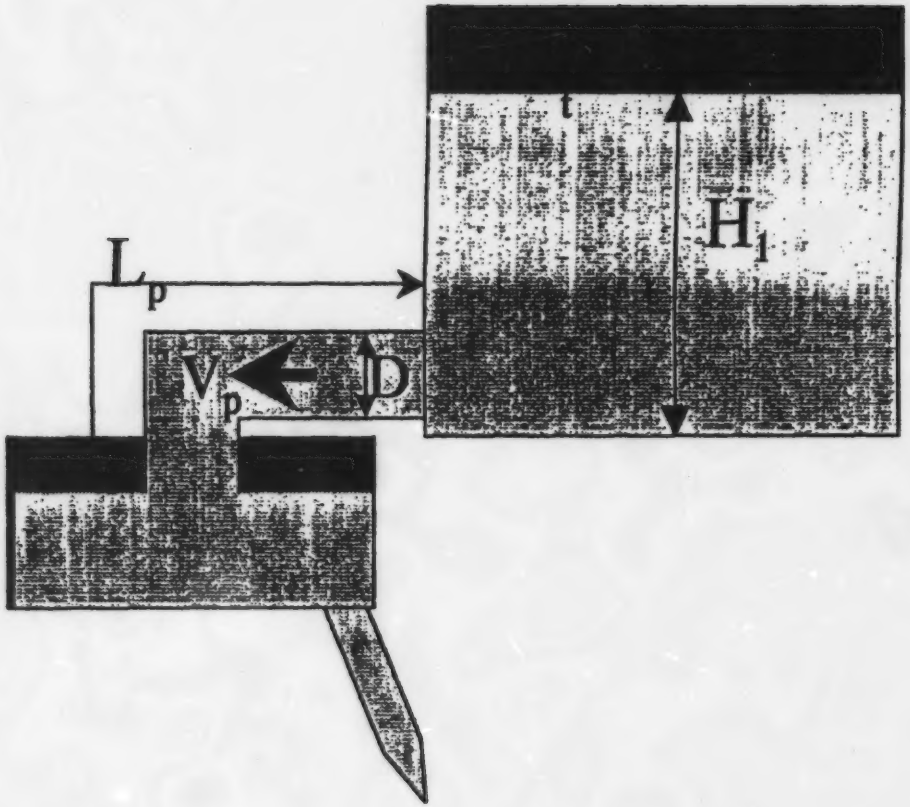


Figure No. 17: Slurry Flow Model

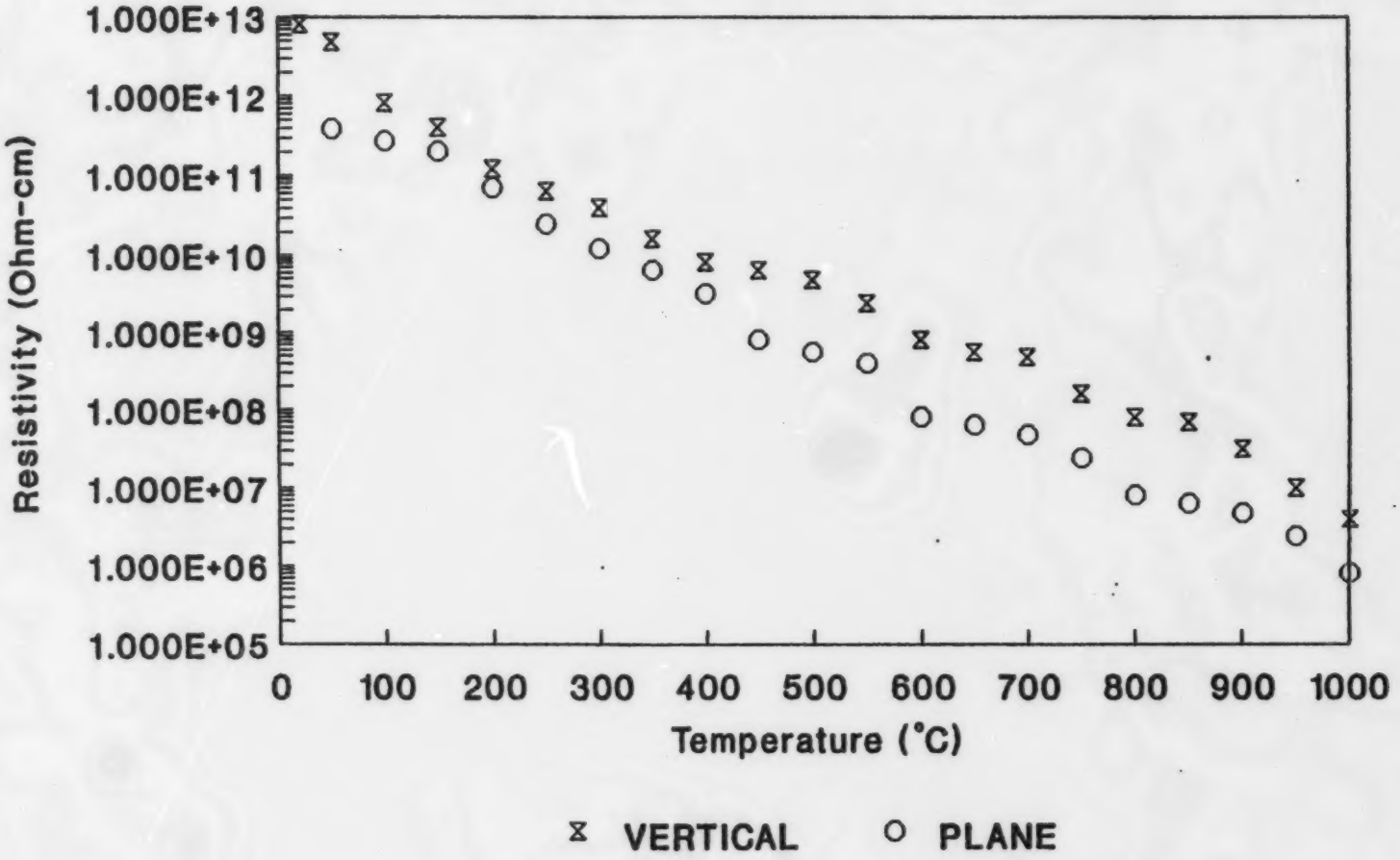


Figure18.Resistivity-Temperature Data for 80 wt.%KAOWOOL-20 wt.%BN

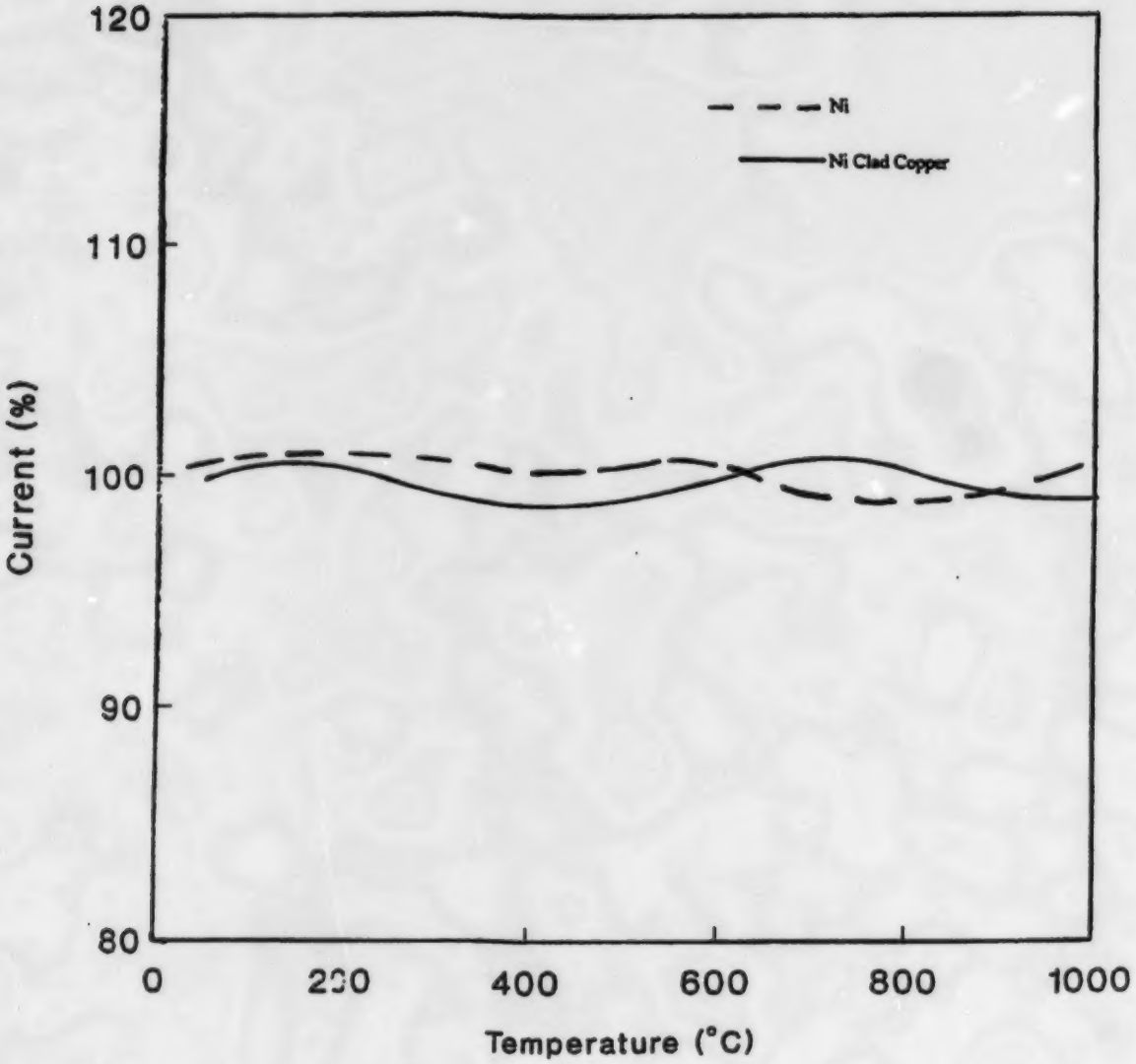


Figure 19. Effect of Temperature on Current in Ni and Ni-clad Copper Cable Segments

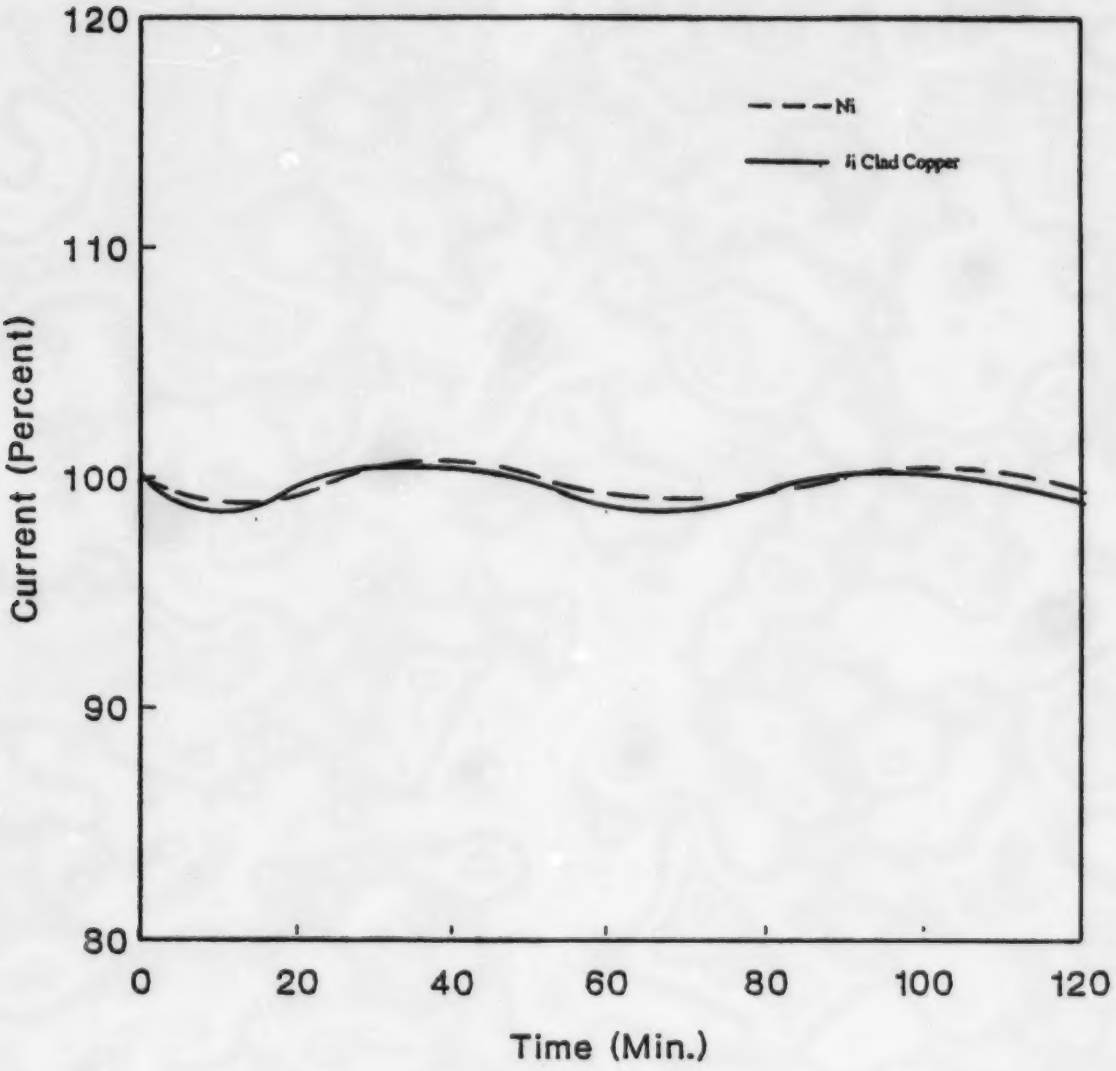


Figure 20. Current in Cable as a Function of Time at 1000 °C .

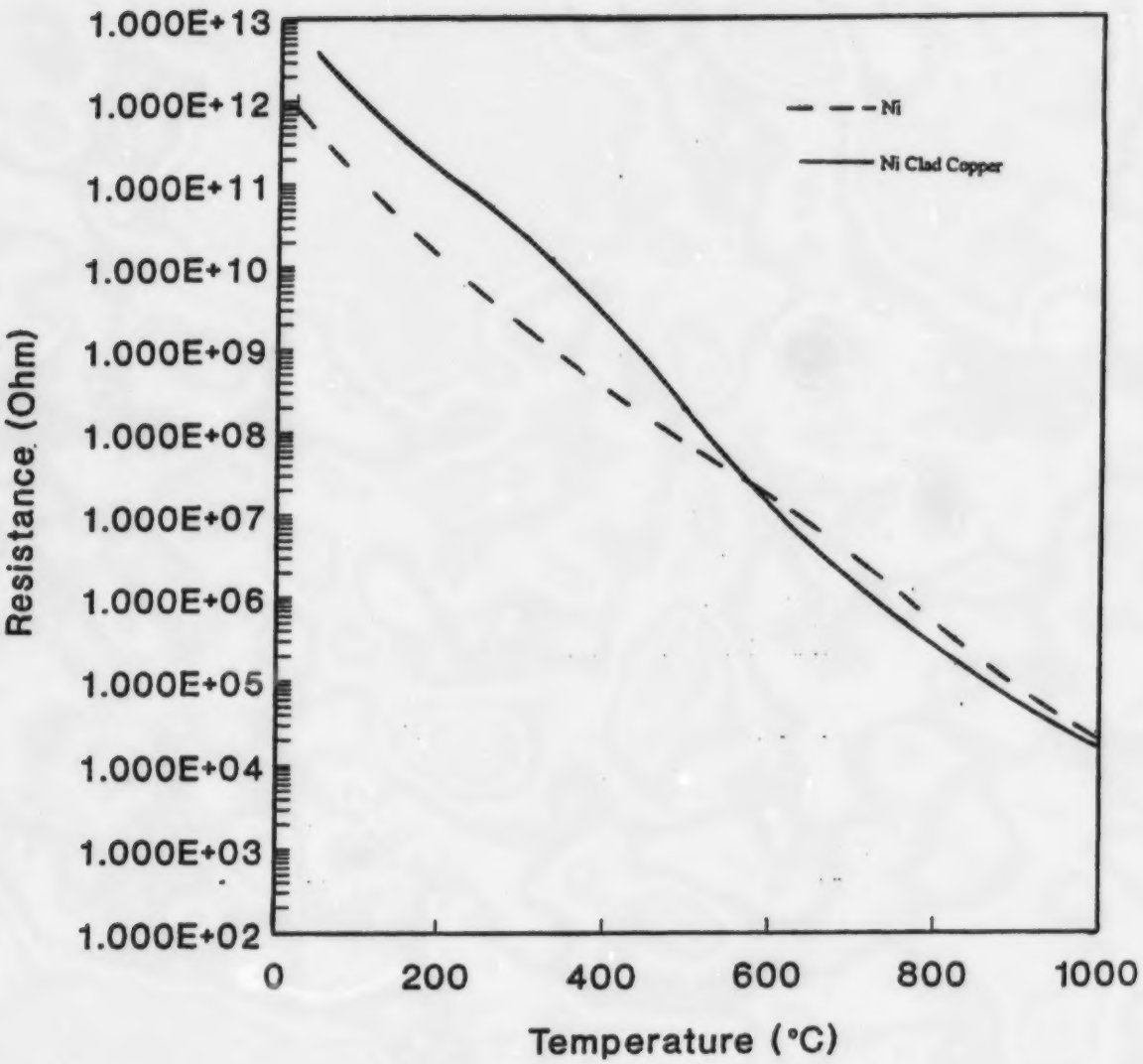


Figure 21. Effect of Temperature on Insulation Resistance (ohm) per cm with Ni and Ni-clad Copper Wires

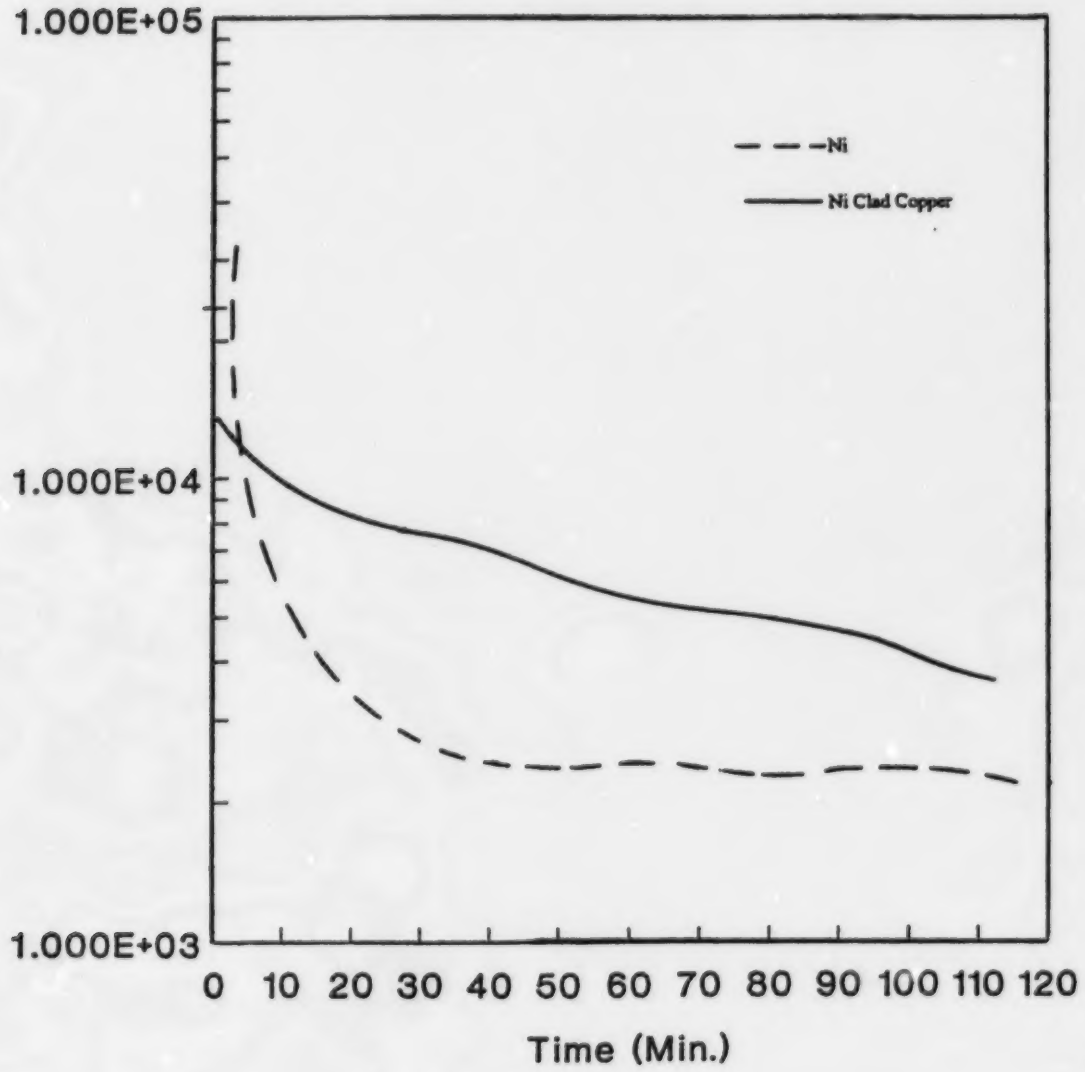


Figure 22. Insulation Resistance(ohm) per cm as Function of Timeat 1000 °C with Ni and Ni-clad Copper Wires

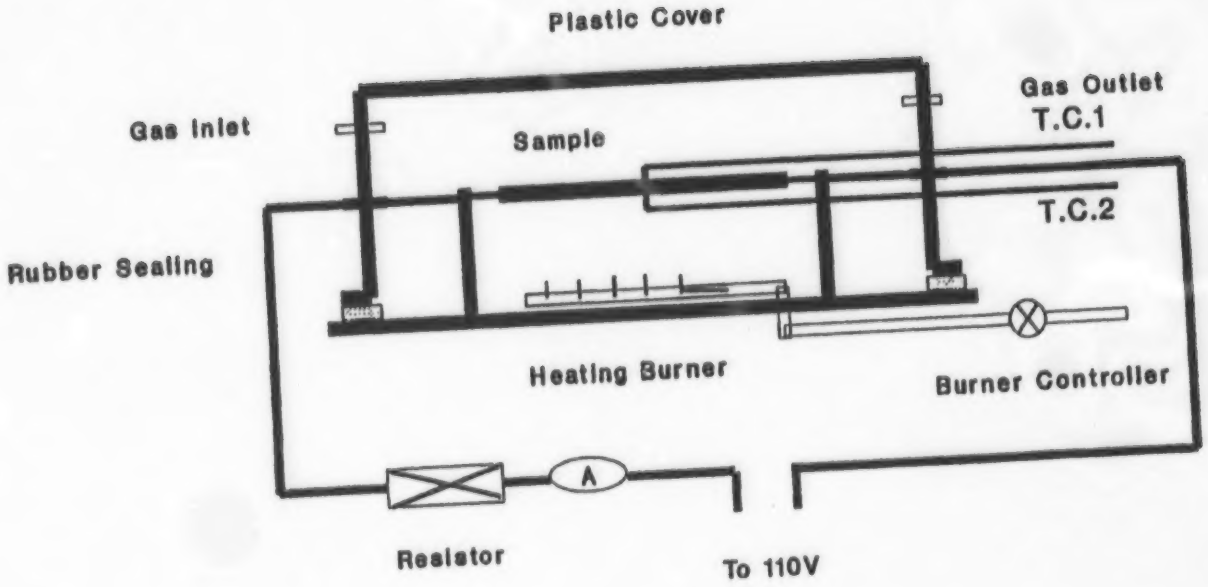
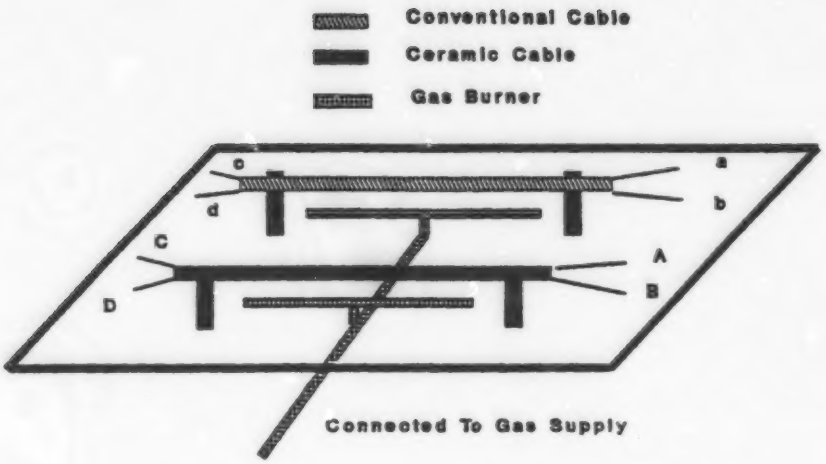
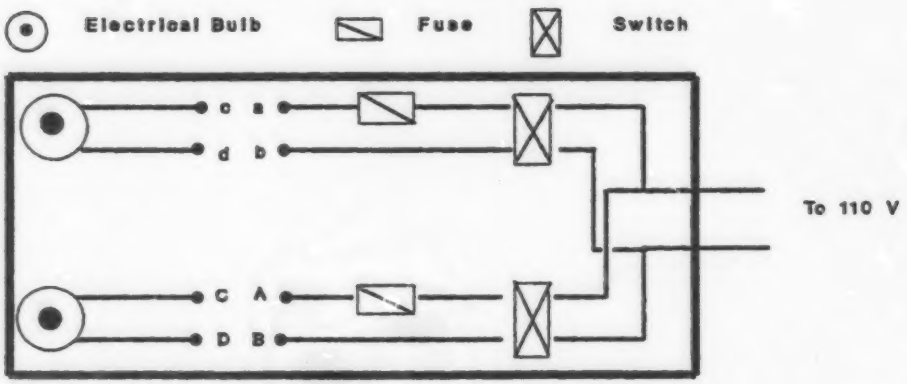


Figure 23. Single Burner Furnace for Burning Tests



(a) Flame Test Chamber



(b) Control Panel

Figure 24. Two Burner Furnace for Burning Tests

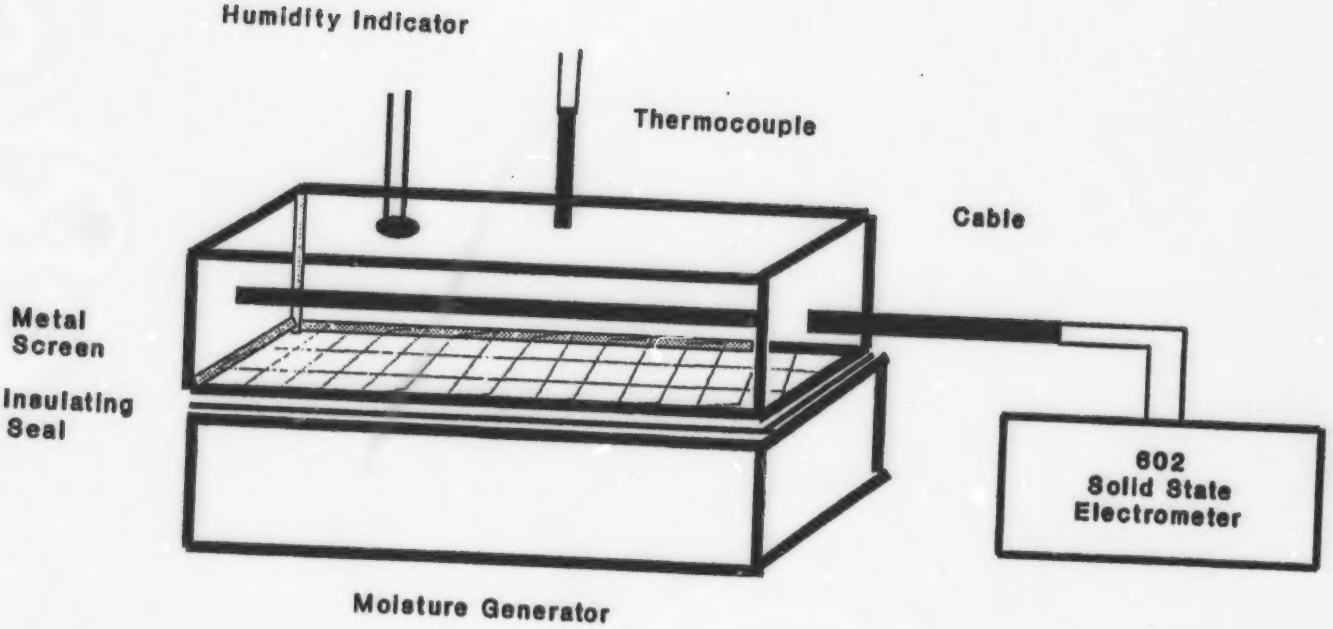


Figure 25. Schematic of Moisture Test Facility

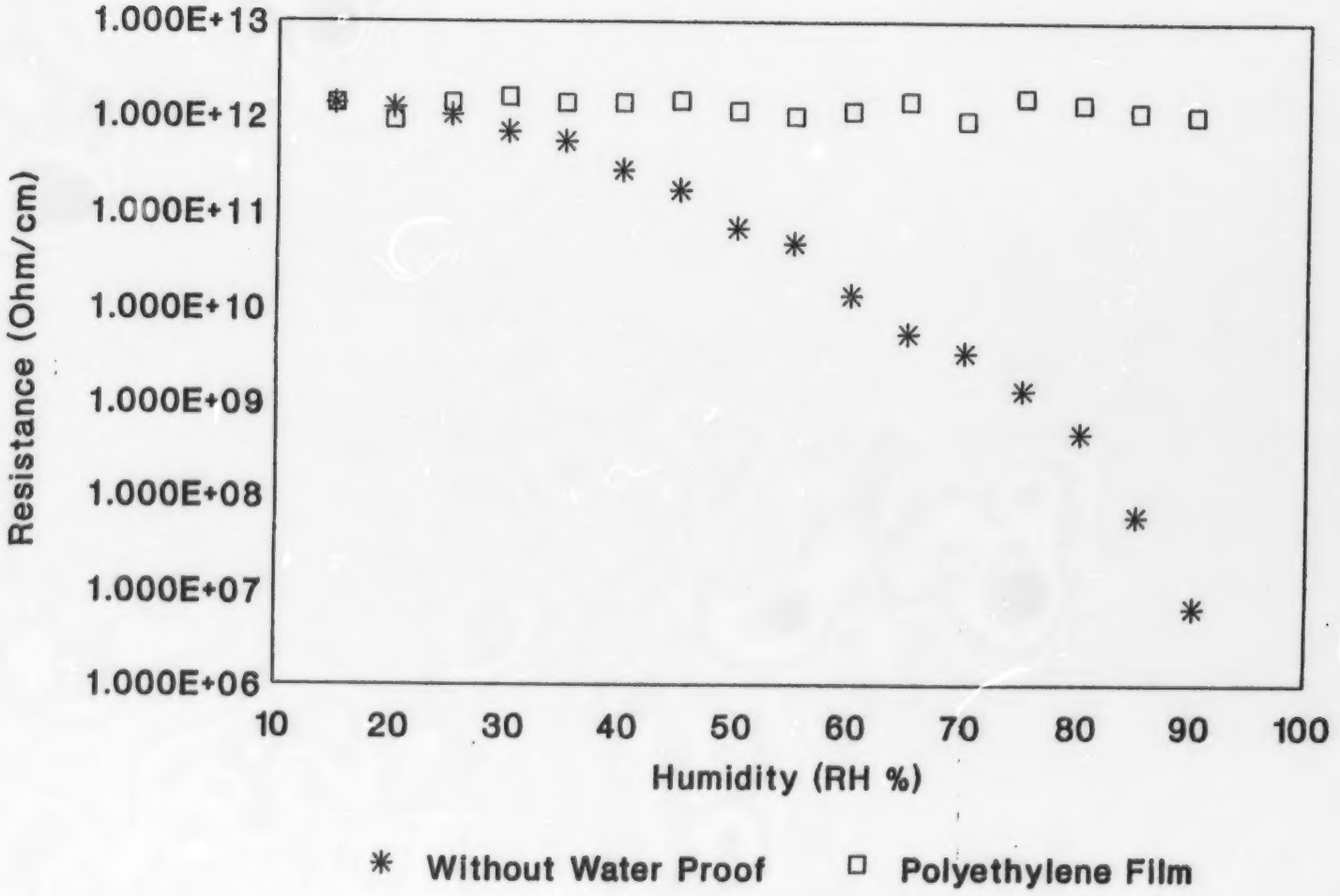


Figure26. Insulation Resistance between Electrodes as a Function of Relative Humidity

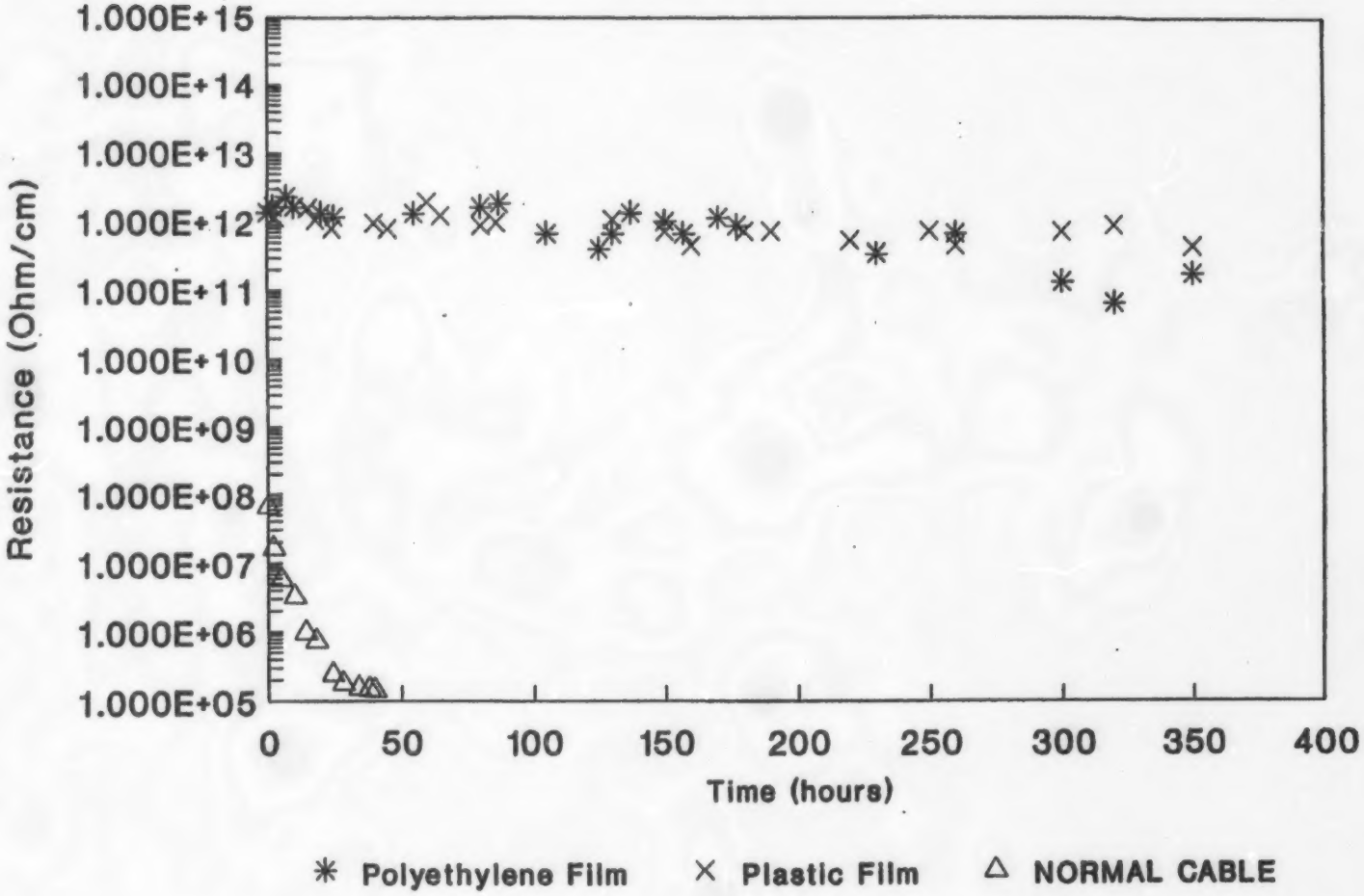


Figure 27. Insulation Resistance between Electrodes as a Function of Time at 85% Relative Humidity

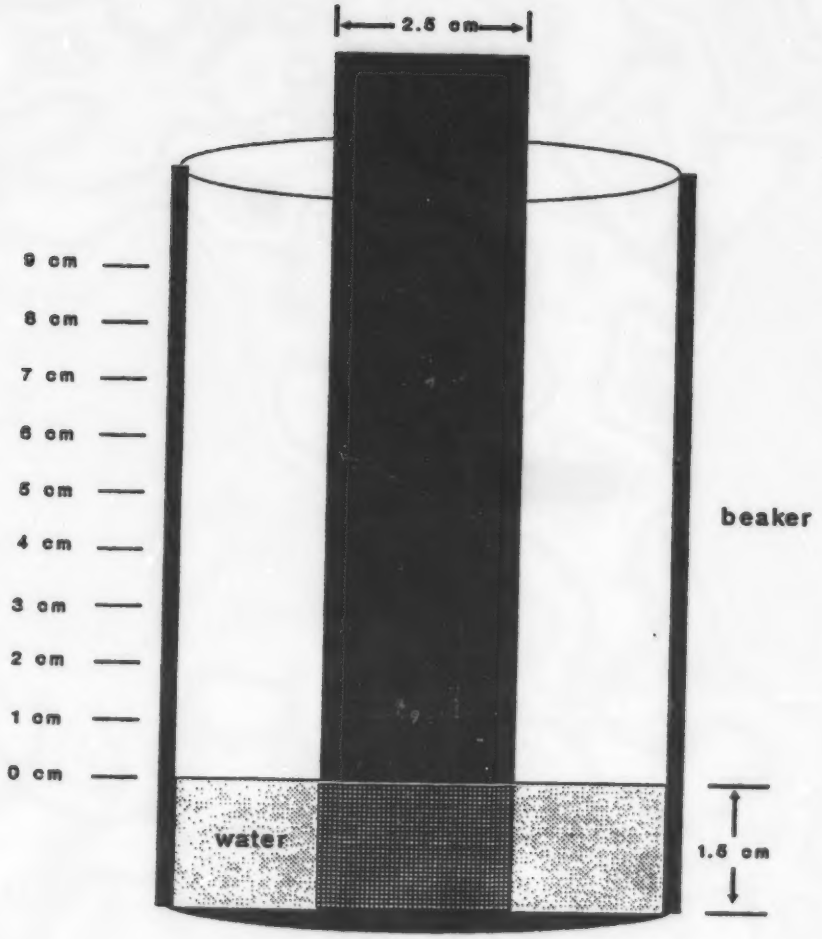


Figure 28. Schematic of Testing Capillary Effect in Ceramic Felts

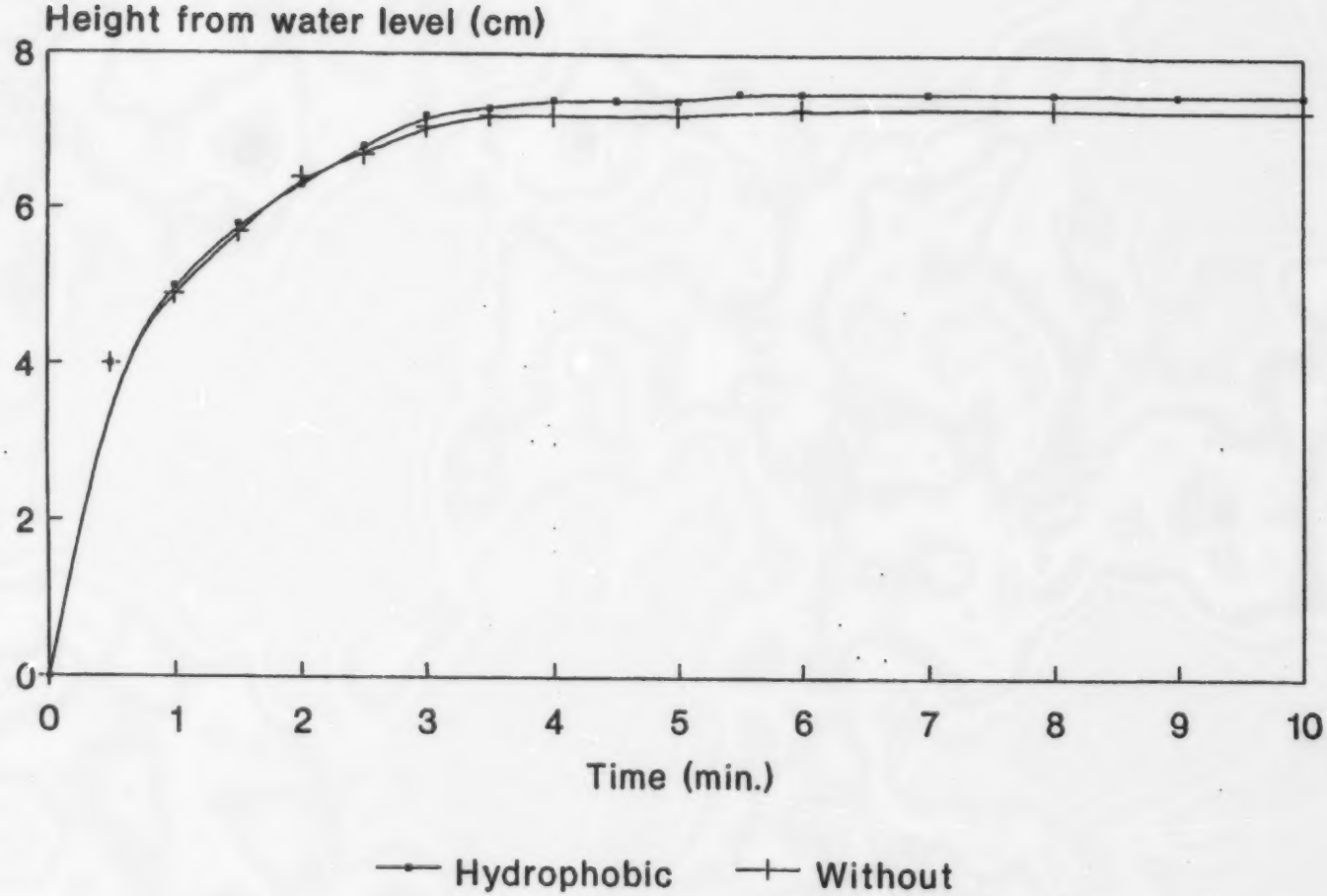


Figure 29. Capillary Effect in Ceramic Felts with and without Hydrophobic Treatment

Figure 30 SEM Micrograph of Ceramic Felt Showing Fiber Distribution

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A cable design has been developed which meets not only the electrical performance requirements, but also is compatible with physical property specifications, e.g., flexibility, mechanical strength, and moisture resistance.

A material composition comprising 80wt% KAOWOOL-20 wt% BN fibers has been developed to meet the property requirements of the electrical insulation of the cable. A unique semi-automatic "slurry-tape-casting" process has been developed for the fabrication of the ceramic insulation in the form of a continuous tape. Prototype cable segments, with lengths up to 12 feet were assembled from the insulation tape using Ni and Ni-coated copper electrical conducting wires. Although manual techniques were used for prototype cable fabrication, engineering concepts were developed for continuous assembly.

Prototype cables have been evaluated for their electrical performance, stability, mechanical properties, and moisture resistance. They have satisfactorily met all their target specifications. They have operated under electrical load at 1000°C for more than one hour. Also, they have withstood open-flame burning tests for more than three hours.

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